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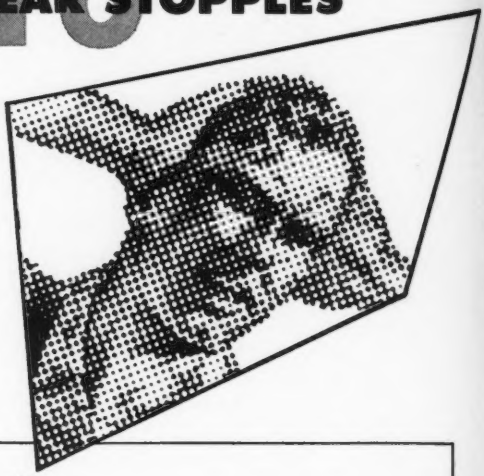

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President's Page

Our Twentieth Annual Meeting and participation in the 1959 Industrial Health Conference held at Chicago, April 25 to May 1, 1959, now are history. The final session for official representatives of the five participating associations was scheduled Friday morning, May 1, to organize committees for planning the 1960 Conference to be held at Rochester, New York, April 25 to April 29. Herbert T. Walworth, a past president of our Association, will serve as General Chairman of the Conference and Franklin A. Miller will serve as chairman in charge of the overall Conference arrangements.

At Chicago, there were many indications that ours is a mature profession and a mature technical society. From the crowded program of 109 technical papers to the presentation of service awards to 19 past presidents at the annual banquet; from the corridor conversations to the agenda of the annual business meeting, there was a general response which represented profound interest and concern for our objectives, our current activities and our continued growth. Although there never has been a lack of interest or support on the part of most of our members, the increasing numbers in our membership and of those attending the annual meetings emphasize reactions to the planning and direction of our Association's affairs by your Board of Directors.

On this page in previous issues, and in News Letters, you have been informed of the activities of the Joint AIHA-ACGIH Committee on Certification Standards. At the annual business meeting on April 29, members present voted overwhelmingly to submit to the membership the current recommendations of the Joint Committee. Details of the plan were included in the News Letter of March 26 and will accompany the ballot which you will receive shortly. The fate of the certification program as proposed by the Joint Committee rests in your hands.

It is unfortunate that not all of our members could be on hand at Chicago for the presentations of Henry F. Smyth, Jr., Ph.D., Chairman of the Joint Committee, and Charles D. Yaffe, Secretary-Treasurer of the ACGIH. Essentially, the presentations concerned highlights of the proposed program and the discussion of questions raised by members of AIHA and ACGIH either in writing in response to the March 26 News Letter or raised from the floor at the session in Chicago. Insofar as practical, the details of the program which will accompany the ballot will reflect consideration of some of the discussion resulting from these questions. The Board of Directors urges your careful study and support of the proposal.

Elmer P. Wheeler

AMERICAN

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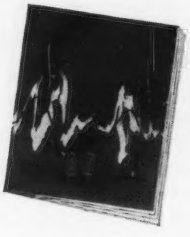

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The Determination of Alpha Activity Associated with Sub-Microgram Quantities of Enriched Uranium in Urine*

T. C. WHITSON, B. S., and T. KWASNOSKI, B.S.

Technical Division, Oak Ridge Gaseous Diffusion Plant, Union Carbide Nuclear Company, Oak Ridge, Tennessee

IN THE INDUSTRIAL hygiene monitoring program carried on at the Oak Ridge Gaseous Diffusion Plant, both the toxicological and radiological aspects of uranium exposures are of interest. Exposures to large amounts of natural uranium may have toxicological effects whereas exposures to high activity enriched material could produce radiological effects because of the alpha emitter uranium-234. Uranium determination in urine does not provide information as to the alpha activity, and since exposure to different assay levels is possible, a separate measurement of alpha activity is necessary.

The alpha activity in enriched uranium is principally due to the uranium-234 which has a specific alpha activity of 1.345×10^7 as compared to 3.824×10^3 and 7.42×10^2 d/min/mg for uranium-235 and −238, respectively. A typical curve showing alpha activity of enriched uranium as a function of uranium-234 content is presented in Figure 1; actual counts at approximately 50% geometry are used in this plot and throughout the remainder of this paper. Since the alpha activity of uranium to which employees may be exposed may vary from 0.75 counts/min/μg to 70 counts/min/μg, it is often possible to predict the area of exposure from the relative uranium and alpha activity determinations on urine.

The plant allowable limit for alpha activity in urine is 1.3 counts/min/100 ml. This limit has been calculated from the *National Bureau of Standards Handbook 52* limit of 0.009 microcuries for the accumulation of insoluble uranium in the lungs. Assuming a 120-day retention period in the lungs, the urinary excretion rate would amount to 26 μg of natural uranium per 24 hours (1500 ml), or an equivalent alpha activity of 1.3 counts/100 ml of urine. While on natural uranium the 1.3 counts would correspond to 1.7 μg U/100 ml, on >90% enriched material this would be equivalent to only 0.016–0.019 μg U/100 ml, depending on the amount of uranium-234 in the enriched material.

Several extraction and precipitation methods for the separation of uranium alpha activity in urine prior to counting have previously been developed. Fletcher[1] used a di-ethyl ether solvent extraction technique with a subsequent evaporation onto a platinum plate. Hurd and Fox[2] employed the coprecipitation of uranium and aluminum with ammonium hydroxide to concentrate the uranium; the dissolved precipitate was evaporated onto a silver disk. Campbell and associates[3] used a solvent extraction with di-n-butyl orthophosphoric acid in carbon tetrachloride; the organic phase was evaporated onto a platinum plate. These methods required extensive operator time in the final evaporation step and frequently left uneven deposits on the counting plates; low recovery and poor precision resulted.

Collecting uranium onto a disk for counting purposes by electrodeposition has a long history. Smith[4] described a method for electrodeposition of uranium onto platinum as early as 1879. Cohen and Hull[5] employed such a method for preparing disks for isotopic counting. McAuliffe and associates[6] investigated electroplating variables and applied the Cohen and Hull procedure to the electrodeposition of uranium onto nickel disks instead of platinum.

This paper concerns itself with a modification of McAuliffe's method that, combined with a prior separation of calcium, has been applied to the determination of alpha activity associated with submicrogram quantities of enriched uranium in urine.

Procedure

The electrodeposition procedure is as follows: one hundred milliliters of urine is evaporated to dryness in a 400 ml beaker with 50 ml of concentrated nitric acid. The residue is ignited to a white salt over a gas burner. The residual salt

* This document is based on work performed at the Oak Ridge Gaseous Diffusion Plant operated by Union Carbide Corporation for the U. S. Atomic Energy Commission. Presented at the Twentieth Annual Meeting of the American Industrial Hygiene Association, Chicago, Illinois, May 1, 1959.

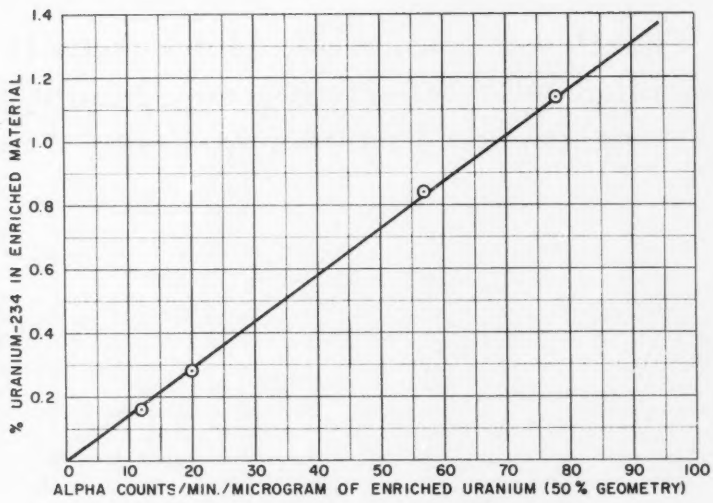

FIGURE 1. Relationship of Alpha Activity of Enriched Uranium to its Uranium-234 Content

is dissolved in five milliliters of concentrated nitric acid and refluxed for a short time to collect any material which may have spattered onto the sides of the beaker. The sample is then taken to near dryness to remove any residual chlorides in the salts. Five milliliters of distilled water is added and the sample again taken to near dryness to remove excess free acid. The residue is dissolved in 10 ml of distilled water and transferred to a 50-ml centrifuge tube. Ten milliliters of 0.4*M* ammonium oxalate is added to the sample in the centrifuge tube and the sample is adjusted to pH 9.6 with ammonium hydroxide using thymol blue as the indicator.

The sample is centrifuged for five minutes and the supernatant liquid containing the uranium as the oxalate complex is poured into an electrolysis cell assembly. The calcium oxalate precipitate is redissolved in a few drops of nitric acid; 5 ml of ammonium oxalate is added and the pH is again adjusted to 9.6 with ammonium hydroxide. The sample is again centrifuged and the second supernate is added to the first in the electrolysis cell asssembly.

Figure 2 shows the cell assembly consisting of a cell stack, base plate, electropolished nickel disk, rubber gasket, and rubber bands.

The electrolysis cell is assembled by placing the electropolished nickel disk into the smaller recessed portion of the steel base plate, fitting the rubber gasket around the plastic cell stack and placing it into the larger recessed area of the base plate. The cell stack is secured to the base plate with four strong rubber bands. This arrangement gives a constant film area and centers the electrodeposit on the nickel disk. The cell assembly is placed on the cell support in the water bath of the multiple plating unit. This unit is shown in Figure 3.

By means of a motor and an endless dental belt, the four pulleys drive the stainless steel shafts and the rotating platinum anodes; when the shaft is in the lowered position, it is rotated by the contact it makes with the pulley. When it is held in the raised position by means of a spring clip, it is free from the pulley and does not turn. Electrical contact with the rotating anode is made through a nickel wire in the mercury reservoir in the top of the shaft. Amperlite tubes provide a current of three amperes.

For the electrolysis, the anode, attached to the stainless steel shaft, is lowered into the electrolyte in the cell assembly and rotated at 500 rpm. The solution is plated at three amperes for two hours. Ten milliliters of 0.4*M* ammonium oxalate is added after one hour to replenish the oxalate ion. Distilled water is added from time to time to maintain the liquid level over the anode. At the end of the plating period, the anode is raised and the current is turned off. The spent electrolyte is discarded. The cell is disassembled; the disk is wiped with paper tissue and ignited for ten min-

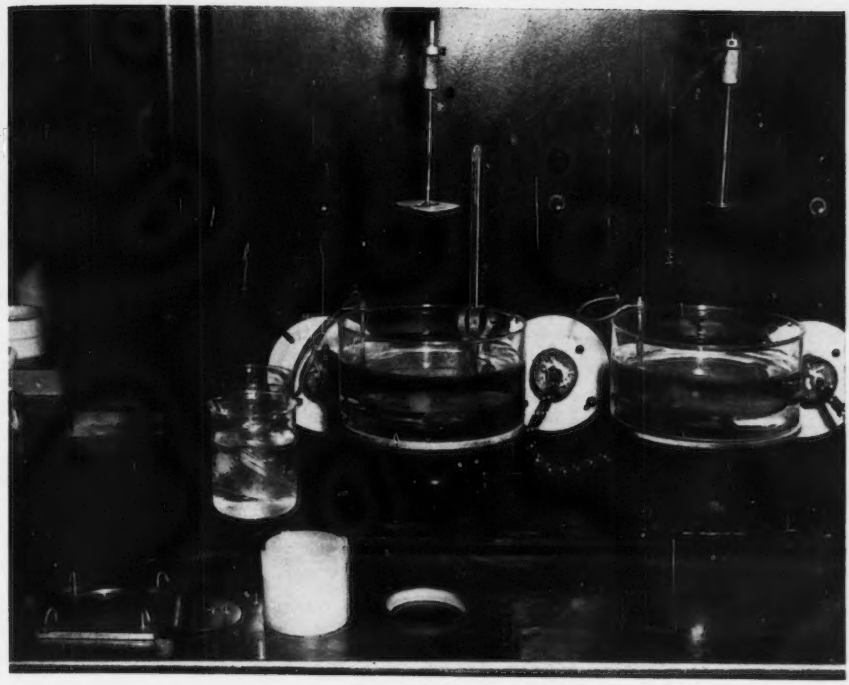

FIGURE 2. Electrolysis Cell Assembly

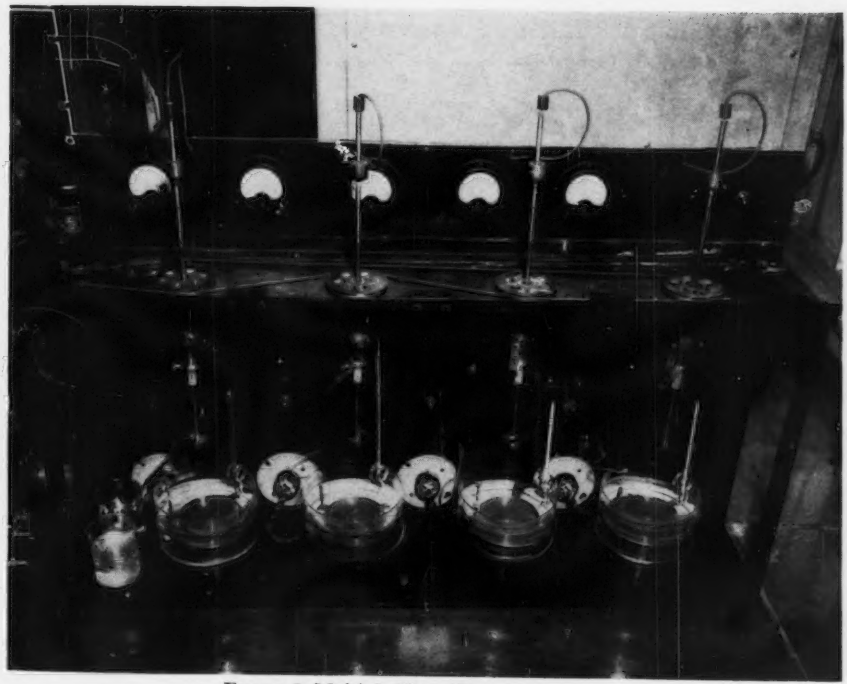

FIGURE 3. Multiple-Place Electroplating Unit

utes at 425°C. The disk is counted for 20 minutes on each of two parallel plate counters.

Discussion

Oxidation of Organic Material: Electrodeposition of uranium from raw urine provides a very simple method for the estimation of alpha activity; however, electrodeposition alone results in the recovery of only 40% of the available activity. Oxidation of the sample with nitric acid, followed by ignition of the residue to a white salt, serves to destroy the organic complexes completely. Dissolution of the white salt in nitric acid converts all of the uranium to the hexavalent state. Recoveries of alpha activity through such oxidation are increased to 62% as shown in Table I.

Oxalate Precipitation: Although oxidation of organic material resulted in an increased recovery of alpha activity, it was evident that even a greater recovery could be realized by the elimination of the heavy salt deposit which formed on the plated uranium film. Calcium was evidently depositing as the oxalate as the pH of the electrolyte increased with plating time. Elimination of this desposit was accomplished by separation of the calcium as oxalate at pH 9.6 before electrolysis. The voluminous amount of precipitate

TABLE I

Direct Electrodeposition of Uranium Tracer from Urine

Plating time: one hour
Sample: Urine + 1300 counts/min (10 μg uranium)

Sample No.	counts/min
1	758
2	999
3	758
4	908
5	758
6	628
7	876
8	701
	Avg. = 802
	Recovery = 62%

TABLE II

Improved Recovery of Uranium Activity from Urine Resulting from Calcium Removal

Plating time: one hour
Sample: Urine + 130 counts/min (1 μg uranium)

Single precipitation	Double precipitation
101	125
115	125
94	124
103	121
87	87
Avg. = 100	Avg. = 116
Recovery = 77%	Recovery = 89%

TABLE III

Quality Control by Procedure for Microgram Quantities of Uranium

Plating time: 40 minutes
Type disk: Unpolished nickel
Control solution: 100 ml urine + 8 μg natural uranium (6 counts/min)

Control period	% Recovery	Absolute LE per analysis (95% confidence level)
January–March, 1957	88	±2.9
April–June, 1957	87	±2.1
July–September, 1957	82	±1.6
October–December, 1957	87	±2.0
January–March, 1958	85	±1.8
April–June, 1958	83	±1.7

TABLE IV

Recovery of Tracer Activity from Water by Procedure for Microgram Quantities of Uranium

Plating time: 40 minutes
Type disk: Unpolished nickel
Sample: Distilled water, ammonium oxalate + 145 counts/min activity (0.016 μg uranium)

Sample No.	counts/min
1	75
2	89
3	32
4	107
	Avg. = 76
	Recovery = 52%

TABLE V

Recovery of Tracer Activity from Water by Procedure for Sub-Microgram Quantities of Uranium

Sample No.	Water + 160 counts/min one hour plating time	Water + 138 counts/min two hours plating time
1	151	139
2	139	143
3	154	136
4	152	135
5	157	130
6	126	145
7	122	145
8	163	134
	Avg. = 146	Avg. = 138
	LE_X = ±11	LE_X = ±13
	Recovery = 88%	Recovery = 100%

which resulted suggested that a second precipitation be performed to minimize the losses of uranium by coprecipitation. Table II shows the results obtained on electrodeposition following single and double precipitation. The recovery of 89% realized by the double precipitation step justified its inclusion in the routine procedure.

Electropolished Disks: Electropolished disks are employed routinely for precision plating of uranium for fission counting because the absence of peaks and valleys on the highly polished surface serves to minimize self-absorption of the alpha count. While our data did not indicate the use of polished disks improved our analytical recovery, they are included in the procedure because of their availability and because of the improved precision which resulted from their use in other applications.

Plating Time: Forty minutes of electrodeposition is adequate for reproducibly recovering about 85% of the alpha activity from microgram quantities of uranium in urine, as shown in Table III, and is the normal plating time in our procedure for microgram quantities of uranium. However, the recovery of sub-microgram quantities (0.016 μg.) of uranium from water after 40 minutes of electroplating is only about 50% as indicated in Table IV; for this study uranium-233 tracer was used to provide a higher alpha count for the small amount of the uranium in the sample. Electrodeposition of the sub-microgram quantities of uranium from water for one hour resulted in nearly 90% recovery whereas essentially complete recovery was achieved in two hours, as indicated in Table V. While the recovery of sub-microgram quantities of uranium from urine after two hours of electrodeposition presented in Table VI is significantly poorer than for the samples electrodeposited from water, it is comparable to that obtained at the microgram level in the routine 40-minute electrodeposition; the losses in either case are attributable to mechanical losses in the separation and possibly to interference in the electrodeposition from ions not separated.

Cross-Contamination: The problem of cross-contamination is a serious one when working at low alpha levels with specimens which cover a wide range of both uranium content and alpha activity. Carry over of activity from one sample to another is minimized by strict adherence to practices of immaculate cleanliness. Where cross-contamination is suspected, additional cleanliness precautions are taken such as the use of all new glassware for each determination.

Precision: At lower counting levels more representative of the urine samples of interest, the precision is affected to a greater degree by the precision of counting. Thus for sub-microgram quantities (0.016 μg. uranium and 1.3 counts/min) the data of Table VII shows a precision of ±0.8 counts/min; the counting precision at this level is estimated to be ±0.6 counts/minute.

TABLE VI

Recovery of Tracer Activity from Urine by Procedure for Sub-Microgram Quantities of Uranium

Plating time: two hours
Type disk: Electropolished nickel
Sample: Urine + 145 counts/min

Sample No.	counts/min
1	127
2	105
3	135
4	101
5	103
6	117
7	138
	—
	Avg. = 118
	LEx = ±38
	Recovery = 81%

TABLE VII

Recovery of Activity from Enriched Uranium in Water and Urine by Procedure for Sub-Microgram Quantities of Uranium

counts/min Water media samples	counts/min Urine samples
1.2	1.5
1.5	1.1
1.0	1.7
1.5	1.4
1.9	1.2
1.0	0.9
0.9	0.7
—	0.8
Avg. = 1.3	1.1
LEx = ±0.8 counts/min	0.5
Recovery = 100%	1.6
	1.0
	—
	Avg. = 1.1
	LEx = ±0.8
	Recovery = 85%

Summary

In summary, a procedure has been developed for routine determination of alpha activity resulting from the presence of sub-microgram quantities of enriched uranium in urine. Wet oxidation of the organic content, removal of the calcium, and two hours of electrodeposition have been found necessary prior to the alpha counting. The method provides over 80% recovery of the alpha

activity with a precision of about ±0.8 counts/minute.

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AS THE FIRST ACTION of his new office, Elmer P Wheeler, President of AIHA for 1959, (right) presented a plaque expressing the Association's appreciation to Kenneth W. Nelson, Past President, who had but a moment before relinquished the gavel of office to Mr. Wheeler. The scene was the dinner meeting of the Twentieth Annual Meeting of the American Industrial Hygiene Association.

Vapor Phase Chromatography in Quantitative Determination of Air Samples Collected in the Field*

R. H. MANSUR, M.S., R. F. PERO, B.S., L. A. KRAUSE, M.S.

Bureau of Industrial Hygiene, Virginia Department of Health, Richmond, Virginia

The Problem

A GOVERNMENTAL industrial hygiene agency is beset by many practical problems. It is constantly called upon to appraise the health hazards of working environments by reasonably valid techniques of measurement, but, for practical reasons, it may not spend a great amount of time on each such appraisal. The cost factor, shortages of trained personnel, the need for rendering prompt service, and other considerations, have led to a demand for and extensive development of numerous direct reading, field instruments.

For direct, field measurement of organic solvent vapor concentrations, three types of instruments are well known; combustible vapor indicators, aromatic hydrocarbon detectors, and halogenated hydrocarbon indicators. These instruments have been, and will continue to be, of great assistance in every-day industrial hygiene activity. Many agencies have, however, frequently been embarrassed by the lack of specificity of these various instruments. For example, workers may be exposed on the job to a mixture of two or more solvent vapors, only one of which may have sufficient toxicity to be of concern. Use of the combustible vapor type of direct reading instrument would give little indication of the health hazard potential of the situation because it would read the total vapor concentration rather than that of the component of principal concern. In certain situations, similar difficulties arise in the use of the other types of field instruments.

The industrial hygienist in the field is frequently burdened with the transportation of numerous pieces of equipment of varying degrees of portability. It would, therefore, be helpful if necessary air sampling equipment could be reduced to a minimum number of items of convenient size, simplicity of operation, and ready availability.

The accuracy of most determinations of solvent vapor concentrations in air has been open to question. This applies to the more complicated and time consuming chemical and physical methods as well as the direct methods.

The problem, therefore, may be stated as follows: To develop a method of quantitative determination of solvent vapors in air that is accurate, rapid, completely specific, and employs a single uncomplicated air sampling technique.

Approach to the Problem

Vapor phase chromatography has become a well accepted tool in many laboratories for a variety of qualitative and quantitative determinations. It has been found especially helpful because of its ability to partition complex mixtures of organic components whose physical and chemical characteristics are so similar that other methods of separation and identification are subject to gross error.

C. D. Lewis, et al.[1] demonstrated the usefulness of a vapor phase chromatography technique for identifying and quantitating trace contaminants in organic solvents in the range of 1–200 ppm. The success of the technique developed by this group encouraged the development of the method described in this paper.

A Perkin-Elmer Model 154B Vapor Fractometer in connection with a Leeds and Northrup Speedomax Type G recorder was acquired. In brief, the principle of operation of the instrument is as follows: Helium is used as the carrier gas, and is fed to the instrument from a pressure bottle at an adjustable pressure and flow rate. A liquid sample is injected into the gas stream where a flash heater almost instantly vaporizes it. The gas-vapor mixture then passes through a packed stainless steel partitioning column which is maintained at a pre-determined and rigidly controlled temperature. The various components of the sample pass through the packed column at different speeds so that, in general, separation is complete or nearly complete by the time the

* Presented at the Twentieth Annual Meeting of the American Industrial Hygiene Association, Chicago, Illinois, May 1, 1959.

sample exits from the column. At this point there is a thermal conductivity sensing cell which detects the passing of a sample component by temperature change between a reference thermistor and a sensing thermistor. The signal thus developed is amplified and transmitted to the five millivolt Speedomax recorder. An attenuator is provided so that the amplified signal may be reduced at will by factors of from 1 to 512. The various components of the sample are recorded on the chart as a series of peaks. Identity of the component is established by the time required to pass through the column, and the percentage of the component is determined by comparing the area under the specific peak with the total area.

When the instrument was first placed in operation, it was used to identify and quantitate the components of liquid samples of such materials as lacquer solvents, cleaning solvents, and paint removers. Using a two meter "A" column as supplied by the manufacturer, it was found that ordinary laboratory grade methanol was given up by the column very early, and that it was relatively free from trace contaminants in the area of interest. This suggested that methanol might be used as the collecting liquid in midget impinger samples for any other solvent that would have a longer retention time in the column.

Accordingly, a 3.7 cubic meter chamber was constructed in which known concentrations could be established for the study of this possibility. The chamber was lined with aluminum foil to make it as nearly gas-tight as possible. The air sampling equipment was located inside the chamber so that error in concentration due to dilution was at a minimum. Known concentrations of various solvent vapor systems were set up by pipetting calculated amounts of liquid solvent material into a platinum dish. The dish was then transferred as rapidly as possible to a small hot plate inside the chamber and the chamber was sealed. After sufficient time for a fan, located in the chamber, to accomplish complete dispersion, the air sampling equipment was started. Controls for the air sampling equipment, fan, and hot plate were all located outside the chamber. Throughout the study, a sampling time of ten minutes at 0.1 cubic feet per minute was used and the sample was collected in 10 ml of methanol. After sampling, the remaining volume of methanol varied between 5 and 6.5 ml. The variation in loss of methanol by evaporation did not appreciably affect reproducibility of final results. As will be shown later, the method is empirical and eliminates the need for correction factors, including the problem of efficiency of collection.

Between 29 microliters and 49 microliters of the impinger samples, collected as described above, were introduced into the Fractometer by means of ordinary hypodermic syringes through the rubber septum provided for that purpose. After considerable preliminary experimentation, it was found that a column temperature of 112°C, a gas pressure of 18 psi, and a gas flow of 56 cc per minute gave measurable peaks on the recorder chart, for most common solvents at any chamber concentration above 15 ppm.

Samples were injected into the instrument with the attenuator set at 256. As soon as the peak representing methanol had passed, 108 seconds from the time of injection, the attenuator was set on 1, or the greatest sensitivity available.

Results

The authors at first believed that the area under a peak for any given sample would vary in direct proportion to the amount of sample injected into the instrument. It followed that it should be possible to use the methanol, in which the sample was collected, as an internal standard. Then, for a given chamber concentration, the ratio of sample peak area to methanol peak area should equal a constant. This was found *not* to be the case.

To illustrate, chamber concentrations of 50 ppm, 100 ppm, and 200 ppm of toluol were set up and impinger samples were collected. When these samples were injected into the Fractometer, the amount injected was purposely varied so that the area of the toluol peak could be plotted against the area of the methanol peak. Figure 1 represents such a plot.

The reader will note that each plot on Figure 1 is essentially a straight line for the range of sample increment sizes injected. If each of these plots were extended, it is seen that they would intercept the vertical coordinate a considerable distance above the zero point. In theory, possibly, the slope of the plot might change with smaller sample portions injected so that the zero point would be met. However, it was found to be practical to inject sufficient sample so that the methanol peak was between 90 and 150 mm. in height in order to obtain peaks of measurable height for the solvent sampled.

Measurement of peak areas by use of a planimeter is time consuming and exacting. It was noted that such measurements, expressed in hundredths of a square inch, were of a similar order of magnitude to the peak heights expressed in millimeters. It was felt, therefore, that comparable accuracy could be attained by using peak height relationships rather than peak area relationships.[2] Subsequent checking indicated that

this was so, even when peaks for certain compounds were not symmetrical in shape. The authors do believe, however, that the method described by Rosie and Grob,[3] where peaks were cut out and weighed on an analytical balance, would be a worthwhile improvement.

Figure 2 shows plots of toluol peak heights against methanol peak heights for chamber concentrations of 50, 100 and 200 ppm. Obviously, it is very similar to Figure 1. It can be seen that, if a valid chart of the type shown in Figure 2 can be developed for each of the solvent vapors usually encountered, it is readily possible to determine a field sample by plotting several in-

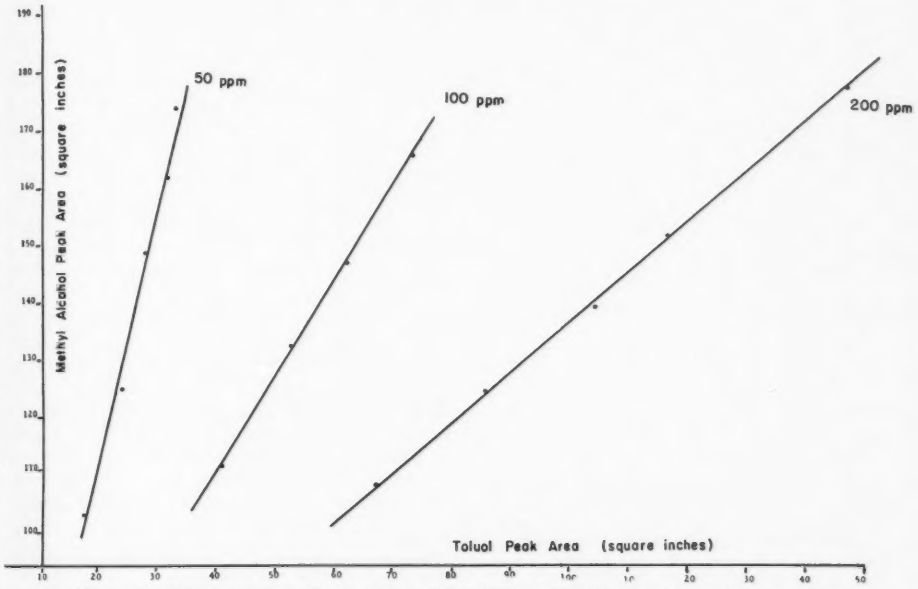

FIGURE 1. Relationship of the peak areas for toluol and methyl alcohol at three concentrations of toluol and with varying amounts of sample injected into the Fractometer.

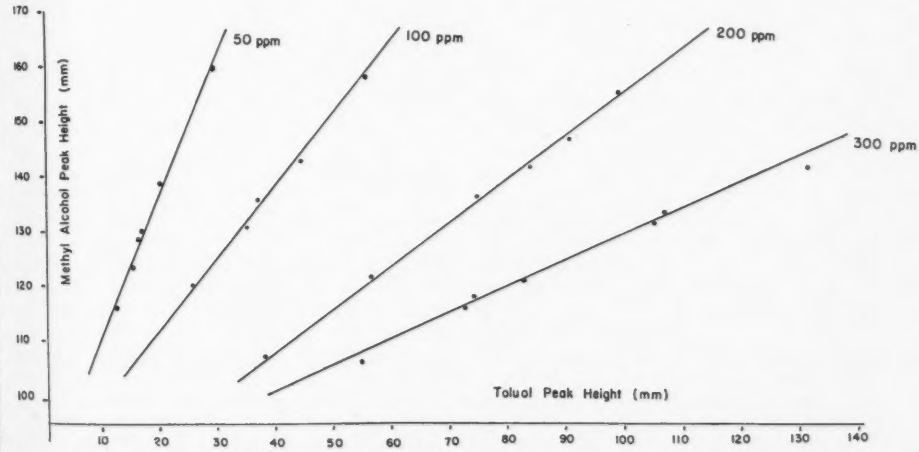

FIGURE 2. Relationship of peak heights for toluol and methyl alcohol at four concentrations of toluol and with varying amounts of sample injected into the Fractometer.

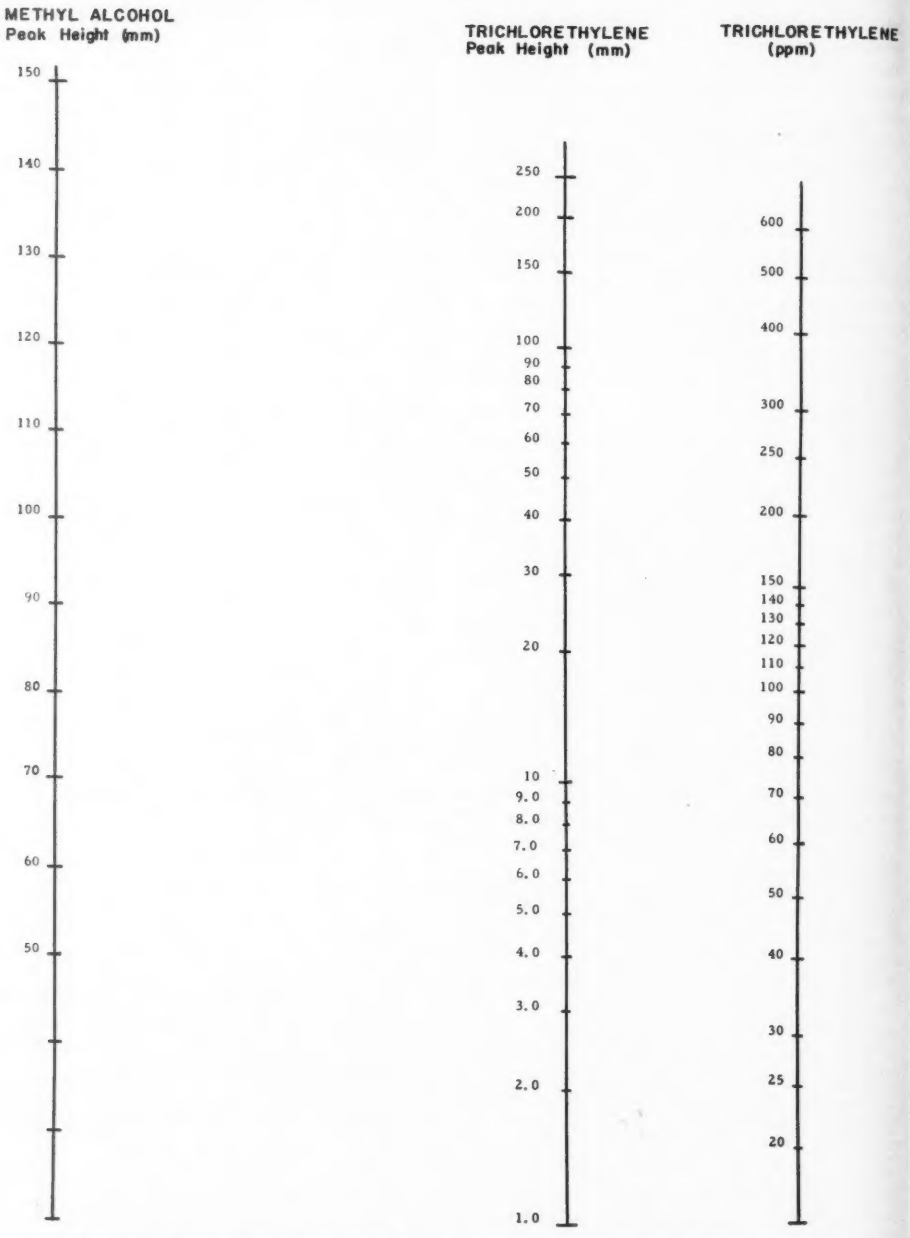

FIGURE 3. Nomogram for the calculation of trichlorethylene concentrations collected in methyl alcohol.

strument runs from it on the appropriate chart or charts, and arrive at a result by interpolation. This has actually been done for benzol, toluol, methyl ethyl ketone, n-butyl alcohol, n-propyl actetate, trichloroethylene, and carbon tetrachloride.

The validity of these charts has been checked by a "blind" sampling technique. One member of the investigating team has set up a large number of chamber concentrations known only to him. Another member of the team has sampled and determined these concentrations as described. Comparison of determined values with the known values indicated a high degree of accuracy.

Charts of the type shown in Figure 2 are somewhat cumbersome, especially if extended to the 400 ppm range and beyond. Furthermore, it was thought desirable to eliminate the necessity for interpolation. Accordingly, an effort was made to develop nomographs for each of the compounds previously listed so that results could be read directly. Figure 3 represents such a nomograph for trichloroethylene. It is seen that the first scale is arithmetic and represents the methanol peak height in millimeters. The second scale is logarithmic and represents the trichloroethylene peak height in millimeters. The third scale, of course, represents the trichlorethylene concentration in air in ppm. This scale was plotted from actual experimental results. As may be seen, it is close to a logarithmic scale of unvarying interval.

Nomographs for other compounds could be shown. However, it suffices to say that the nomograph for each compound appears to have its own unique characteristics, dependent on volatility of the compound, heat transfer coefficient, column retention time, solubility in the collecting methanol, and possibly other factors. Validity of the nomographs was tested by the "blind" sampling technique previously described. Comparison of determined results with known values again indicated a high degree of accuracy (Table I).

It was sometimes found that attempts to validate charts or nomographs that had been prepared a considerable time previously did not give results of quite the desired accuracy. It is not known exactly what causes the change in degree of accuracy at certain times. There may be changes in conditions of instrument operation which have not yet been observed. There is reason to believe, however, that extreme changes in the moisture content of sampled air may be the major factor.

Fortunately, there is a ready means of resolving this difficulty. It is not unduly time consuming to set up a known concentration in the chamber to be sampled and determined at the same time a set of field samples is to be run. Several instruments runs of the known sample are read on the appropriate nomograph. If an error due to changed conditions is apparent, the average deviation from the proper value on the nomograph scale is measured with a metric scale. Then, the results obtained on the field samples are adjusted by the same linear distance on the nomograph scale.

This method was again tested by a "blind" sampling technique. It was found that the maximum error on the basis of one instrument run was ±10%. If three to five instrument runs are used, the error is consistently within ±5% at all concentrations studied. This degree of accuracy is considered fully adequate for practical industrial hygiene use. Table I tabulates a few typical results obtained by this testing method.

The laboratory time required for determina-

TABLE I

Determinations of Known Concentrations of Toluene by Chromatography Using Nomograph Reading

Chamber concentration (ppm)	Corrected nomograph reading (ppm)	% Error
50	48.6	2.5
50	49.8	.5
50	49.8	.5
50	46.4	7.0
50	48.6	2.5
	Average	2.6%
100	104.0	4.0
100	106.0	6.0
100	103.0	3.0
100	105.0	5.0
100	103.0	3.0
	Average	4.2%
150	155	3.3
150	155	3.3
150	156	3.7
150	155	3.3
150	157	4.5
	Average	3.4%
200	197.6	1.5
200	198.4	1.0
200	195.0	2.5
200	199.0	0.5
200	196.0	2.0
	Average	1.5%
300	276	8.0
300	280	6.5
300	288	4.0
300	290	3.0
300	285	5.0
	Average	5.3%

tion of field samples is approximately as follows: one hour to one and one-half hours for each setup of known concentrations with subsequent sampling and five instrument runs, plus one hour for each field sample including three to five instrument runs. During the course of the study, the authors were called upon to do determinations on three field samples for tetrachlorethylene, a substance not previously studied. The total elapsed time in the laboratory required for development of complete background data and determination of the three samples was less than one day.

It should be understood that these are several errors inherent in the method prior to the time of actual injection of the sample into the Fractometer. These include errors in pipetting small amounts of liquid solvent, loss by evaporation before placing them in the chamber, variations in sampling rate in the chamber, slight leakage of the chamber, and the known errors of the static chamber method of setting up known concentrations. It is believed that the sum of these errors may be as great as the apparent overall error stated above.

Figure 4 illustrates three types of recorder tracings obtained during the study. Figure 4A shows a toluol peak representing a concentration in air of 200 ppm and the methanol peak. The methanol peak was recorded with the attenuator set at 256, and the toluol peak with the attenuator set at 1. It is seen that the methanol peak tracing does not soon return to zero with an attenuator setting of 1. The "tailing" is probably caused by non-ideal adsorption of the components on the surface of the support.[4] It would, of course, be desirable to use a collecting solvent that was not polar and would be more completely resolved by the column. Further study may involve a more suitable material if it can be found. Meanwhile, it can be shown that the error produced by the long methanol tail is not serious[3] and the drift is gradual enough to permit successive runs after the base line is again adjusted.

An extension of the methanol tail is drawn with a French curve, and the toluol peak height is measured from tip to methanol tail as indicated by the dotted line. Except for the presence of the methanol tail, the toluol peak has a satisfactory symmetrical shape.

Figure 4B illustrates an extreme condition. The peak at the left represents methyl ethyl ketone at 125 ppm. It is seen that it rests very high on the methanol tail so that its shape is far from symmetrical. Nevertheless, the methanol tail extension was drawn in with a French curve and the MEK peak height was measured vertically from its tip to the methanol tail. Exhaustive study has shown that, although its relationship to peak area is questionable, this height measurement technique enables reproducible and accurate results. It can be said that, as long as methanol is used as the collecting solvent, the shape of the MEK peak will be essentially the same.

Figure 4C illustrates peaks of two substances which come off the column so near the same time that either interference or enhancement might be suspected. The "m" shaped peak represents n-butyl acetate at 100 ppm and toluol at 100

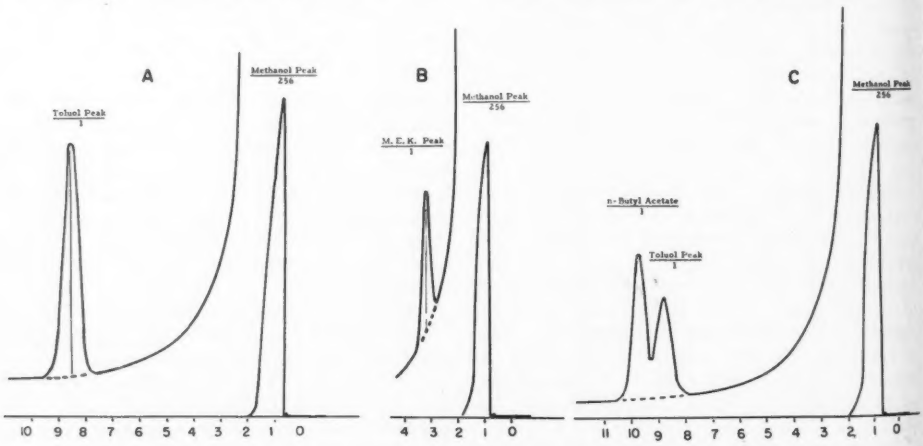

FIGURE 4. Illustrating three types of recorder tracings obtained: A. Toluol at 200 ppm, B. Methyl ethyl ketone at 125 ppm, and C. N-butyl acetate and toluol, each at 100 ppm. Each sample was collected in 10 ml methanol at 0.1 cfm for 10 minutes.

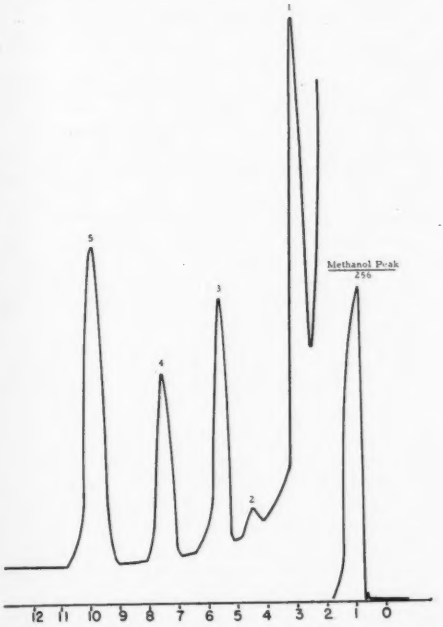

FIGURE 5. Tracing obtained with a five component system sampled in methanol. The components and corresponding peaks are: (1) ethyl acetate, 400 ppm; (2) benzene, 25 ppm; (3) n-butyl alcohol, 100 ppm; (4) methyl iso-butyl ketone, 100 ppm; and (5) n-butyl acetate, 200 ppm.

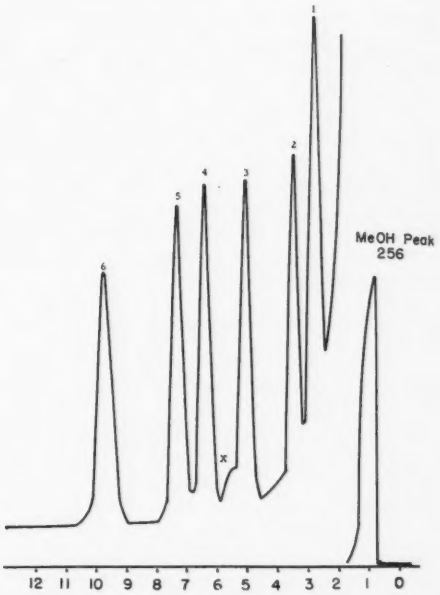

FIGURE 6. Tracing of six related esters sampled at their respective Threshold Limit concentrations: (1) Ethyl acetate, 400 ppm; (2) iso-propyl acetate, 200 ppm; (3) n-propyl acetate, 200 ppm; (4) sec-butyl acetate, 200 ppm; (5) iso-butyl acetate, 200 ppm; (6) n-butyl acetate, 200 ppm; and (X) acetic acid impurity.

ppm. The matter was extensively studied, and it was found that the error introduced by interference or enhancement is relatively minor. If, however, one wishes to obtain greatest accuracy, it is readily possible to set up known concentrations of the mixture in the chamber at slightly higher and slightly lower levels. Peak height relationships for the two knowns and one unknown can then be plotted, and a more accurate result obtained by interpolation.

Figure 5 is a reproduction of a recorder tracing representing a five component solvent vapor system, all at the maximum acceptable concentration in air. Reading from left to right, the peaks represent n-butyl acetate, methyl iso-butyl ketone, butyl alcohol, benzene, ethyl acetate and the collecting methanol. Although the solvent mixture was a synthetic one, the figure shows that quantitative determination of each component of a complex system is possible.

Figure 5 will also serve to illustrate one further point. Note that the ethyl acetate peak representing 400 ppm goes slightly off scale. If the concentration of ethyl acetate had been above the maximum acceptable concentration of 400 ppm, the peak would not have been readable. In such a case, it has been found possible to set the attenuator at 2 until the peak has passed. The reduced peak can then be read on the appropriate chart or nomograph, and the result multiplied by 2. No loss of accuracy by this device has been noted.

A recorder tracing representing six closely related esters, all at the MAC in air, is seen in Figure 6. Reading from right to left the peaks represent the collecting methanol, ethyl acetate, iso-propyl acetate, n-propyl acetate, s-butyl acetate, iso-butyl acetate, and n-butyl acetate. This demonstrates that even the various isomeric forms of organic vapors in air can be separated, identified, and quantitated. The slight peak marked X is interesting in that it represents acetic acid present in the esters as an impurity.

Comment

The opinion has been stated that, "Present gas chromatographic techniques do not have the

sensitivity to determine solvents in air at most threshold concentrations".[5] We believe that the method described in this paper has eliminated this objection.

A widely held opinion has been that sampling methods involving either adsorption on silica gel or activated carbon or freeze out techniques would offer greater promise.[6, 7] The authors believe that such methods, while academically sound, would be unnecessarily cumbersome and time-consuming, and would offer several additional opportunities for error.

Summary

Progress in development of a method of using vapor phase chromatography for the quantitative determination of solvent vapors in air has been described. It is concluded that, in the present stage of development, the method is usable from the standpoints of accuracy, specificity, time requirement, and cost. It is emphasized, however, that certain features of the method should have further study. It is also believed that, with further study by other interested groups, the general principles of the method can be modified to fit numerous other needs of the industrial hygiene profession.

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Engineering Estimates of Atmospheric Dispersal Capacity*

HARRISON E. CRAMER, SC.D.

Massachusetts Institute of Technology, Cambridge, Massachusetts

Introduction

MOST AIR POLLUTION problems of industry involve small-scale atmospheric diffusion processes.[1] Contaminants are discharged within the lowest layers of the atmosphere and are diluted to insignificant levels of concentration after maximum travel distances of about one to 10 miles from the source. Removal of all pollutants at the point of emission through source-control measures is hindered by cost factors and lack of technical knowledge. From a purely pragmatic viewpoint, source control and abatement techniques must be employed to reduce contaminant emission to levels that can be safely handled by the atmosphere. Efficient use of this natural waste-disposal system clearly depends upon reliable estimates of atmospheric dispersal capacity which varies widely in both space and time. Predictions of effluent dilution based on formulas previously available[2, 3] have lacked the degree of precision necessary for effective engineering treatment of the atmospheric waste-disposal problems of industry. This is in part explained by the scarcity of satisfactory measurements.

Recent empirical investigations[4, 5, 6, 7, 8, 9, 10, 11] have significantly increased the fundamental knowledge of small-scale dispersal mechanisms. These studies provide, for the first time, a comprehensive picture of the variations in basic diffusion parameters in all thermal stratifications. In addition, analyses of these data have led to a new technique for estimating the dispersal of airborne contaminants based on the use of direct meteorological indicators. Estimates obtained by this method are believed sufficiently accurate to provide a basis for resolving many industrial air-pollution problems. The purpose of this paper is to summarize these recent developments in air-pollution meteorology and describe their use in obtaining comprehensive estimates of atmospheric dispersal capacity for small-scale processes.

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Small-scale Dispersal Theory

For sampling times of the order of 10 to 30 minutes, the average concentration $\chi_{(y,z)}$ at any distance x downwind from a continuous elevated point source is given by the equation of continuity in the form[5]

$$\chi_{(y,z)} = \frac{Q}{2\pi\bar{u}\sigma_y\sigma_z} \exp\left[-\tfrac{1}{2}(y^2/\sigma_y^2 + z^2/\sigma_z^2)\right], \quad (1)$$

where Q is the source strength or emission rate, $\bar{u}$ is the mean horizontal wind speed; σ_y, σ_z are the standard deviations of concentration along the lateral y and vertical z axes of the plume, respectively; and, x is in the direction of the mean wind. The concentration χ_a at the center of the plume ($y = 0$; $z = 0$) is

$$\chi_a = \frac{Q}{2\pi\bar{u}\sigma_y\sigma_z}. \quad (2)$$

The above expressions, which hold for any consistent system of units, are based on the usual assumptions: the crossplume and vertical distributions of concentration are Gaussian; the mean wind speed is invariant with space and time; and, the plume is not subject to bouyancy forces nor to depletion by gravitational settling. Due to reflection from the earth's surface, the emission rate Q should be doubled for a source at ground-level or in the case of a plume from an elevated source that intersects the ground. The height and width of the plume, defined in terms of the usual 10 per cent limits of axial concentration, are given by 4.3 σ_z and 4.3 σ_y, respectively.

For any given values of source strength and mean wind speed, it is clear that the downwind distribution of concentration depends principally upon σ_y and σ_z. These basic diffusion parameters are highly correlated with the standard deviations of azimuth angle σ_A and elevation angle σ_E measured near the point of emission.[5, 7, 8] For maximum travel distances of the order of one mile and in all thermal stratifications except ex-

treme instability and stability, the distance dependence of σ_y and σ_z is approximately described by simple power laws of the form

$$\sigma_y = \sigma_A x^q; \qquad \sigma_z = \sigma_E x^p;$$

where σ_A, σ_E are in radians and p, q are constants. When the above expressions are inserted in equations (1) and (2), the result is

$$\chi_{(x,y,z)} = \frac{Q}{2\pi\bar{u}x^b\sigma_A\sigma_E} \exp\left[-\tfrac{1}{2}(y^2/\sigma_A{}^2x^{2q-2} + z^2/\sigma_E{}^2x^{2p-2})\right]; \tag{3}$$

and,

$$\chi_a = \frac{Q}{2\pi\bar{u}x^b\sigma_A\sigma_E}; \tag{4}$$

where $b = p + q$ and y, z, σ_A, σ_E are in radians.

The concentration at ground level χ_g (along the line $y = 0$; $z = -h$), due to the intersection with the earth's surface of a plume from a continuous elevated point source at height h, is given by

$$\chi_g = \frac{Q}{\pi\bar{u}x^b\sigma_A\sigma_E} \exp\left[-\tfrac{1}{2}(h^2/\sigma_E{}^2x^{2p})\right]. \tag{5}$$

The maximum ground-level concentration χ_{mg} and the distance x_m from the base of the stack at which it is found are similarly expressed as

$$\chi_{mg} = \frac{Q(b/p)^{b/2p}\sigma_E{}^{b/p-1}}{\pi\bar{u}h^{b/p}\sigma_A} \exp\left[-b/2p\right]; \tag{6}$$

and,

$$x_m = \left[\frac{h^2p}{\sigma_E{}^2b}\right]^{1/2p}. \tag{7}$$

Approximate values for the dispersal parameters in the above equations are presented in Table I. Entries for σ_A in stable stratifications provide

TABLE I

Small-scale Dispersal Parameters for Various Thermal Stratifications

(Entries marked with an asterisk apply strictly at travel distances of the order of 0.5 mile and should not be used for appreciably longer or shorter distances.)

Thermal stratification	σ_A (deg)	σ_E (deg)	b	p	q
Extremely unstable	25–30	8–30	2	1.1	0.9
Moderately unstable	15–20	6–8	2	1.1	0.9
Near-neutral	6–15	3–5	1.85	1.0	0.85
Moderately stable	5–15	2–4	1.6*	0.8*	0.8*
Extremely stable	2–15	0–2	1.3*	0.6*	0.7*

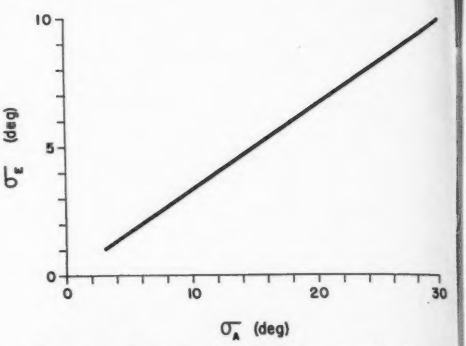

FIGURE 1. Relationship between standard deviations of azimuth angle σ_A and elevation angle σ_E near ground level.

for the long-period meandering frequently observed during temperature inversions. If this factor is not included, the upper limit for σ_A is about four degrees in extremely stable stratification and about ten degrees in moderately stable stratification. With this exception, σ_A and σ_E are quasi-dependent throughout the entire range of stability. The parameter σ_A alone shows significant promise of serving as a universal indicator of dispersal since it contains implicit information on surface roughness and other factors.[4, 5] The relationship between σ_A and σ_E near ground level is presented in Figure 1; the solid line is based on a regression analysis of measurements obtained by the Massachusetts Institute of Technology during Project Prairie Grass. These data lead to a value of about 0.33 for the ratio between σ_E and σ_A. Somewhat larger values of the ratio probably apply at heights of several hundred feet above ground level during extremely unstable stratification; smaller values will usually be found at these levels in stable stratification. It should be pointed out that there are as yet no satisfactory empirical determinations of the exponent p for continuous elevated sources in the presence of extreme instability. Evidence based on sources near ground level[5] indicates that values of $p \gg 1$ should be found at distances of the order of one mile from the source. At short travel distances from an elevated source, a value of $p \approx 1$ appears reasonable. Since there is no empirical evidence that the plume from a continuous elevated source comes to the ground during stable thermal stratification, the following working formulas for χ_{mg} and x_m are obtained from equations (6), (7) and Table I:

$$\chi_{mg} \approx \frac{2Q}{\pi e\bar{u}h^{1.9}}(\sigma_E/\sigma_A); \tag{8}$$

and,

$$x_m \approx h/1.35\sigma_E . \quad (9)$$

These expressions are practically identical with those of O. G. Sutton[2] except for the substitution of direct meteorological indicators for the generalized diffusion coefficients C_y, C_z and the slightly smaller value of the exponent on stack height. The general form of the axial ground-level concentration profile obtained from the above system of equations appears in Figure 2.

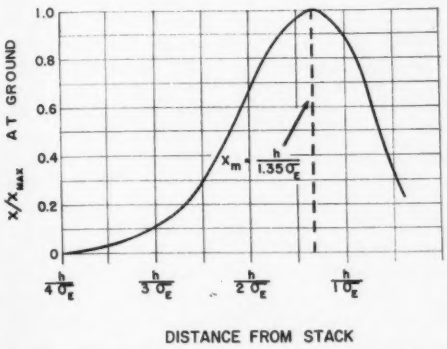

FIGURE 2. Generalized form of axial ground-level concentration profile. Distance from base of stack is given by ratio of stack height to standard deviation of elevation angle (in radians); dashed line indicates distance at which maximum occurs.

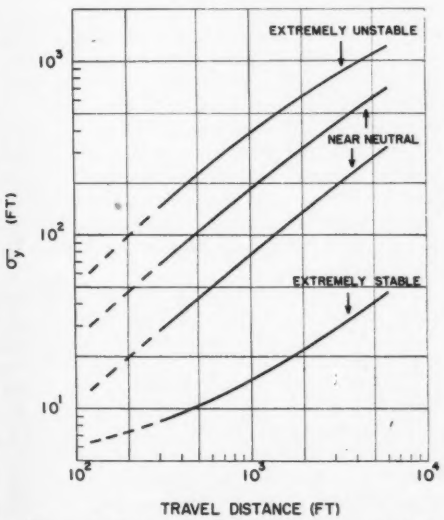

FIGURE 3. Estimates of crossplume standard deviation of concentration downwind from continuous elevated point source in various thermal stratifications. Dashed lines indicate minimum values for area source. Estimates refer to ten-minute sampling time.

Gross Limits of Atmospheric Dispersal Capacity for Small-scale Processes

Specification of the gross limits of atmospheric dispersal capacity begins with calculations of axial concentrations at various distances downwind from a continuous elevated point source in all thermal stratifications. The most direct method involves substitution of appropriate values for the standard deviations of concentration σ_y, σ_z in equation (2). These results do not depend upon the power-law approximations shown in Table I and in equations (3) through (9). Estimates of σ_y and σ_z for extremely unstable, near-neutral, and extremely stable stratification are presented in Figures 3 and 4. These values are based on regression analyses of comprehensive ground-level concentration measurements obtained by the Massachusetts Institute of

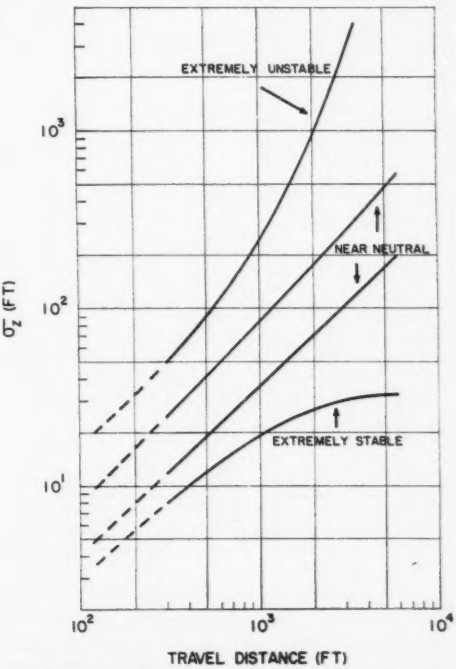

FIGURE 4. Estimates of the standard deviation of concentration along the vertical plume coordinate downwind from a continuous elevated point source in various thermal stratifications. Dashed lines indicate minimum values for area source.

Technology.[4, 5] The σ_z estimates for extreme instability have been altered to conform, at short and intermediate travel distances, to the rectilinear vertical spread (p = 1) anticipated in the case of an elevated source. Two sets of values are presented for near-neutral stratification to provide for differences in surface roughness. This factor is minimized in other stratifications since, in lapse conditions, mechanical turbulence associated with roughness elements is dominated by convective turbulence; and, in stable atmospheres, mechanical turbulence is rapidly damped with increasing height. The larger estimates of σ_y and σ_z in Figures 3 and 4 for near-neutral conditions refer to a rough surface; the smaller estimates refer to a relatively flat surface with few obstructions. In spite of the fact that these figures are based on ground-level measurements, they appear to be entirely consistent with observations so far reported for elevated sources.[9, 10] As mentioned previously, confirmation of these results is lacking only in the case of extreme instability for which the above estimates of σ_z seem conservative. The dashed lines in Figures 3 and 4 indicate minimum travel distances at which the lateral and vertical dimensions of plumes from conventional sources (stacks) will probably exceed the point-source values. Standard deviations of azimuth angle and elevation angle corresponding to the stability classifications in the above figures are presented in Table II.

TABLE II

Values of the Standard Deviations of Azimuth Angle σ_A and Elevation Angle σ_E Associated with the Thermal Stratifications of Figures 3, 4, and 5.

Thermal stratification	σ_A (deg)	σ_E (deg)
Extremely unstable	30	10
Near-neutral (rough surface)	15	5
Near-neutral (smooth surface)	6	2
Extremely stable	3	1

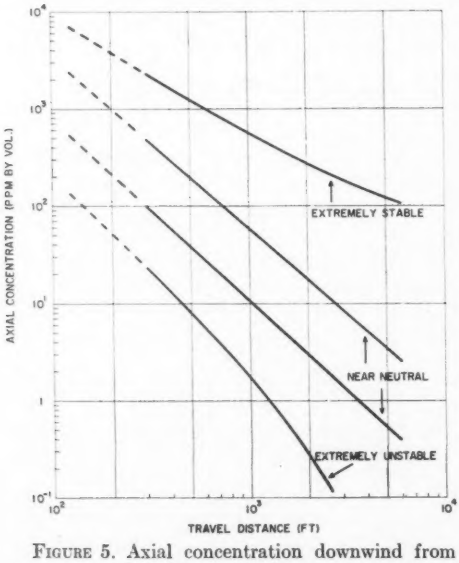

FIGURE 5. Axial concentration downwind from continuous elevated point source in various thermal stratifications. Concentrations refer to ten-minute sampling interval and are adjusted for ratio of unity between emission rate and mean wind speed.

Axial concentrations shown in Figure 5 were obtained from Figures 3 and 4, and equation (2). They represent concentrations on the centerline of a plume from a continuous elevated point source for sampling periods of the order of ten minutes; for convenience, the ratio of the emission rate Q (cfs) to the mean wind speed $\bar{u}$ (fps) has been set equal to unity. Adjustment of these values for sampling intervals of longer or shorter duration is discussed below (see Figure 9). The dashed lines indicate minimum travel distances at which the concentrations based on a point source will likely exceed those for a conventional source. The axial concentrations of Figure 5 are doubled in the case of a source located at ground level. As an example of the interpretation of these results, consider a source with an emission rate of 1800 cfm and a mean wind speed at stack height of 30 fps: According to the figure, the maximum 10-minute concentration at a distance of about 0.5 mile from the stack, and at height h above ground level, will range from 0.1 to 200 ppm depending upon the thermal stratification. For other values of emission rate and wind speed, the concentrations in Figure 5 are multiplied by the ratio $Q/\bar{u}$.

Ground-level concentration profiles (along the line y = 0; z = −h) for extremely unstable and near-neutral stratification have been calculated from equation (1) and the data in Figures 4 and 5. The results for a stack height of 100 ft are shown in Figure 6. Plots of the maximum ground-level concentration and the distance from the base of stack at which it occurs appear in Figures 7 and 8 for stack heights within the range from 50 to 1000 ft. These calculations may be summarized as follows. Estimates of maximum ground-level concentration are about one-third to one-half as large as Sutton's values.[2] The estimates are in close agreement with those of Bosanquet and Pearson[3] for stack heights of 100 to 200 ft; in the case of tall stacks, the Bosanquet

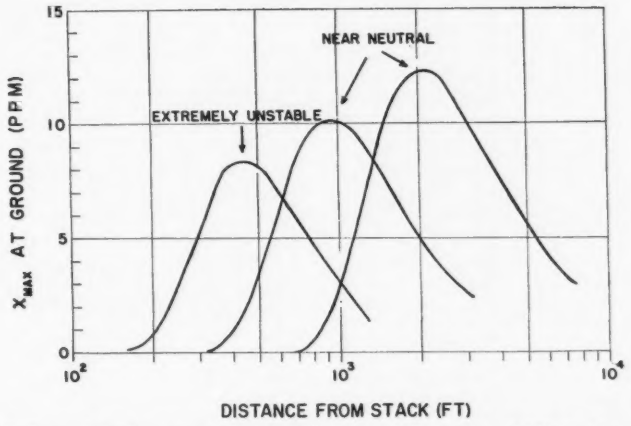

FIGURE 6. Ground-level concentration profiles for stack height of 100 ft. Concentrations refer to ten-minute sampling interval and are adjusted for ratio of unity between emission rate and mean wind speed.

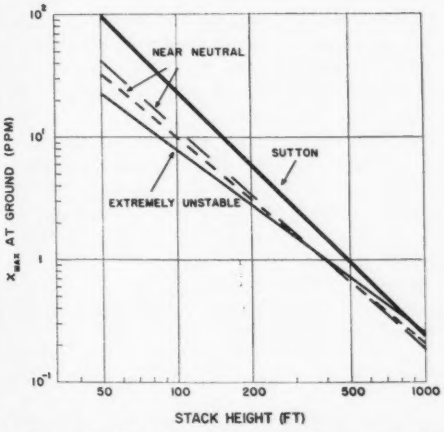

FIGURE 7. Maximum ground-level concentration as a function of stack height. Concentrations refer to ten-minute sampling period and ratio of unity between emission rate and mean wind speed.

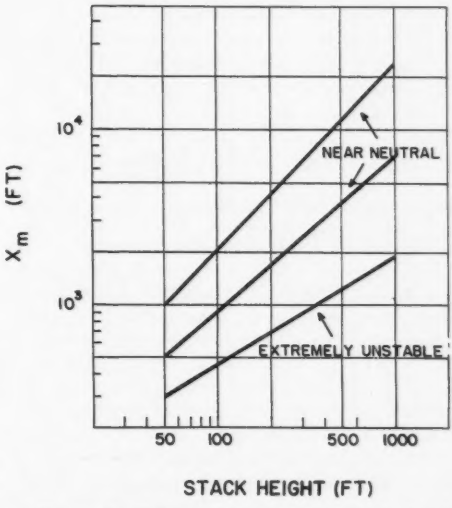

FIGURE 8. Relationship between stack height and distance from base of stack at which maximum ground-level concentration occurs.

and Pearson concentrations are only half as large as the above estimates. The relationship between maximum ground-level concentration and stack height does not follow the inverse-square law usually assumed.[2, 3] Instead, the dependence of χ_{mg} on stack height is approximately defined by a power law in which the absolute value of the exponent is about 1.8 for near-neutral conditions. The calculations also indicate that the value of χ_{mg} is invariant with thermal stratification for stack heights of the order of 300 ft. The profiles of Figure 6 show the variation in maximum ground-level concentration with thermal stratification for short stacks; this pattern tends to be reversed in the case of very tall stacks. The relationship between stack height and the distance from the base of the stack at which the maximum ground-level concentration occurs appears in Figure 8. These results are generally in accord with

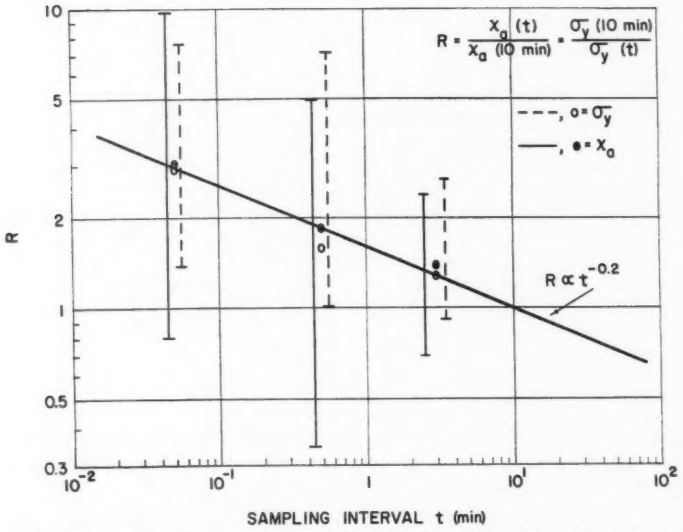

FIGURE 9. Adjustment of ten-minute axial concentrations and standard deviations of crossplume concentration to sampling intervals from a few seconds to one hour. Open and filled circles are averages of Round Hill measurements and vertical lines indicate extremes.[4, 11] Solid line is one-fifth power law reported by Stewart, Gale, and Crooks.[9]

previous estimates i.e., x_m ranges from about 3 h to 20 h depending upon the stratification.

Adjustment of Dispersal Estimates for Variations in Sampling Time

The dispersal estimates discussed above refer to sampling times of the order of ten minutes. They may be adjusted for sampling intervals ranging from a few seconds to one hour by reference to the data presented in Figure 9. The general relationship between concentration and the duration of the sampling interval follows the one-fifth power law reported by Stewart, Gale, and Crooks[9] which is based on measurements at Harwell, England for sampling times from about three minutes to one hour. Recent data obtained by the Massachusetts Institute of Technology[4, 11] indicate that the same power law also applies for sampling intervals from ten minutes to three seconds. As shown in Figure 9, the three-second axial concentrations are generally about three times larger than the ten-minute values; in the extreme (as shown by the vertical lines), this difference may be one order of magnitude. Hourly concentrations are about 70 per cent of the ten-minute values. It also appears that the time dependence of axial concentration is largely explained by variations in the crossplume standard deviation σ_y. This is not entirely unexpected in view of the general form of the power spectra of the eddy velocities.[12, 13] The relationship between σ_y and the duration of the sampling interval shown in Figure 9 cannot be expected to hold precisely at long travel distances and at heights greater than a few hundred feet; however, the absence of vertical meandering in contrast to the wide lateral meandering of plumes suggests that it is qualitatively correct.

Summary

The material presented in previous sections of the paper is intended to provide a suitable base for engineering estimates of small-scale atmospheric dispersal. Many of the numerical results are approximately midway between estimates previously obtained from the formulas of O. G. Sutton[2] and Bosanquet and Pearson.[3] This is interpreted as an indication that considerable confidence may be placed in predictions of the dispersal of airborne material. In general, it is anticipated that calculated values of effluent dilution should be within a factor of two of the values actually observed. No attempt has been made to treat specific applications of the prediction techniques described in the paper; in most instances, this requires the attention of properly qualified specialists in air pollution. The comments following are intended to serve as a

general guide in the use of the formulas and other prediction aids.

It is suggested that values of the mean wind speed and the standard deviations of azimuth and elevation angle near stack height be used in estimating concentrations along the centerline of a plume from a continuous elevated source. For estimates of ground-level concentration, particularly in near-neutral stratification, the mean wind speed and standard deviation of azimuth angle at heights of the order of ten feet above the earth's surface are appropriate. When surface roughness characteristics change significantly downwind from the source, adjustments should be made in the basic dispersal parameters; additional measurements of the mean wind speed and the frequency distribution of azimuth wind direction at one or more points along the plume trajectory provide the most satisfactory basis. When the lateral spread of the plume is limited by topographical features or when the vertical spread is limited by temperature inversions, suitable modifications must be made. For large stack exit velocities and large differences between the exit temperature of the effluent and ambient air temperature, the actual height of the plume centerline may be considerably greater than the stack height. Although no completely satisfactory method for estimating the thermal and velocity rise is available, the technique described by Bosanquet, Carey, and Halton[14] appears to be the the most suitable. The Harwell data[9] indicate that this method rarely leads to an overestimate of effective stack height. Bodurtha[15] has recently discussed the application of this technique in connection with the operation of flare stacks. In calculating ground-level concentrations, it is important to use only about half of the maximum thermal and velocity rise given by these formulas.

There is some question about the necessity for direct measurements of both azimuth angle and elevation angle in routine predictions of dispersal. Although many additional observations are required to resolve this matter satisfactorily, it appears that a knowledge only of the mean wind speed and the frequency distribution of azimuth wind direction suffices for reasonably accurate dispersal estimates.[4, 5]

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The Value of Phase Microscopy for the Examination of Particulate Material Collected on Membrane Filters*

GERMAIN CROSSMON, B.S.

Bausch & Lomb Optical Company, Rochester, New York

A NUMBER of papers have been published suggesting the use of membrane filters such as the Millipore type AA for the collection of dust particles in air. The usual procedure is to place the filter in a holder and sample at a rate of 0.1 to 0.5 cfm using an electrically driven pump or "Freon" powered equipment[1] such as employed in the impinger method. For some applications, First and Silverman[2] find that an adequate sample can be obtained with a few strokes of a 600 ml handpump.

After collection of the dust sample, the membrane filter or a section of it is placed dust side down on a clean slide and a drop of oil added to render the filter transparent so that the particulate material collected can be counted and its particle size determined. The usual method of examination is with the light-field microscope. Drinker and Hatch[3] suggest the use of the standard low power set-up as used with samples collected in an impinger. For maximum resolution, they prefer the oil immersion objective used in conjunction with a calibrated grid in the eyepiece, and give calculations for determining the number of dust particles per cubic foot of air at that magnification.

Another method of sample preparation is to place the filter dust side down on the ruling of a hemacytometer and add a drop of the clearing oil. The hemacytometer coverglass is applied to the preparation and examination made with the 10x, 16mm or 21x, 8mm objective. The depth of focus of these objectives, especially in the case of the 10x, is such that both the ruling of the hemacytometer and dust particles within the filter can be observed.

An objection to the light-field method of examination is the fact that dust particles of small size, having a refractive index close to that of the oil used for rendering the filter transparent, will not be visible or at least difficult to see. This limitation has been indicated by First and Silverman,[2] also by Drinker and Hatch.[3] Paulus, Talvitie, Fraser, and Keenan[4] find that quartz dust of refractive index 1.54 and diatomite with indices of 1.43 and 1.48 will not all be counted with light-field illumination. Their observation should be especially true in the case of calcined diatomite. Calcining of amorphous diatomaceous earth for industrial use results in a conversion to crystalline silicon dioxide (cristobalite) with a resulting increase in refractive indices to approximately 1.484 and 1.487 more closely approaching the index of the clearing oil. They have tried staining the oil placed on the filter with an organic dye to bring out the smaller particles but have had little success. First and Silverman[2] suggest the use of an oil differing slightly from the index of the filter or dark-field counting obtained by use of surface illuminating objectives or a Bausch & Lomb dark-field dust counter.

Our experience with Millipore filters, type AA and HA obtained from the Millipore Filter Corporation have indicated that one solution to the above problem of invisibility of dust particles is the use of phase microscopy. In a previous publication,[5] we have described the superiority of this microscope for the counting of dust particles collected in an impinger and counted in a Sedgwick-Rafter cell, Dunn cell or hemacytometer. The same procedure with necessary modifications applies to the use of the membrane filter. However, in this case, consideration of the fact must be made that the phase microscope is capable of differentiating very slight differences in refractive index in the specimen. According to Chamot and Mason,[6] it gives values to an additional decimal place beyond the usual Becke test as determined by use of light-field microscopy. Cedarwood oil and synthetic immersion oil of index 1.515 that have been suggested for clearing membrane filters prerequisite to counting have too high an index to be successfully used with phase microscopy. This limitation is illustrated in Figure 1 showing calcinated diatomaceous earth particles on an AA type filter in Cargille oil of index 1.515. The degree of difference in refractive

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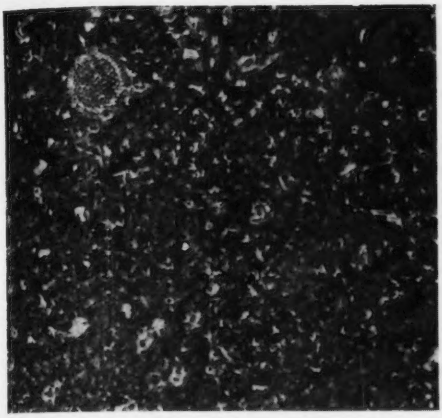

FIGURE 1. Diatomaceous earth particles on a Millipore type AA filter. Cleared in a liquid of refractive index 1.515. Phase microscope × 210.

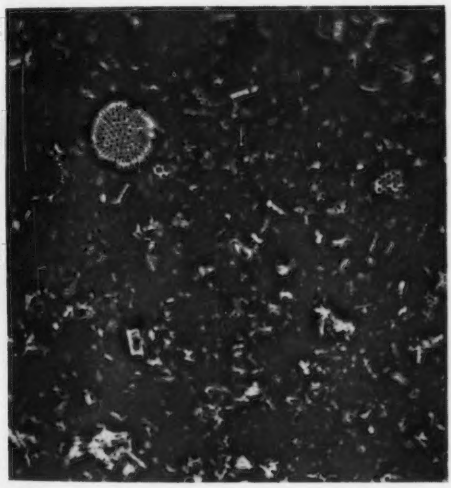

FIGURE 2. Same as Figure 1 but cleared in a 1.507 index liquid. Phase microscope × 210.

index between the filter and the oil is such that the texture of the filter is revealed and might easily be confused with small particle size dust.

Following the above observation, we obtained some additional samples of types AA and HA Millipore filters of more current production and determined their index using the phase microscope with sodium illumination. It was found that the index of the AA type filter was 1.507 at 25°C. The index of the HA type was very similar, any

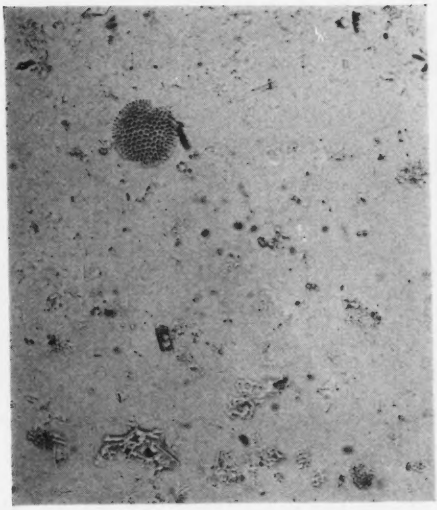

FIGURE 3. Exactly the same area as Figure 2. Cleared in a 1.507 liquid. Light-field microscope × 210.

difference being in the fourth decimal place. Using a liquid of that index, the filter texture is rendered completely transparent as shown in Figure 2 of calcined diatomaceous earth. Figure 3 is of exactly the same field as photographed with light-field illumination. Figures 4 and 5 represent results obtained in the case of small particle size dust consisting mainly of quartz (indices 1.544 and 1.553) as observed by these two methods of illumination.

It is important that the constituents of the 1.507 liquid have no solvent effect on the filter. A mixture of mineral oil with Aroclor 1242 (Monsanto Chemical Compay) adjusted to that index on a refractometer at 25°C has proved to be very satisfactory. Another possibility, if this 1.507 liquid is not available, is isoamyl salicylate (Eastman Kodak Company). It is a colorless liquid with pleasant odor. The sample purchased had an index of 1.506 at 25°C close enough to that of the filter as to result in almost complete transparency. In using liquids for rendering membrane filters transparent, consideration must be made of the fact that the refractive index of liquids varies with temperature. Slight differences are not important but use of a 1.507 liquid corrected for that index at 25°C and used at a much lower or higher temperature may result in visibility of the texture of the membrane filter.

Equipment previously suggested[5] for the counting and particle size determination of dust in counting chambers was the use of the long

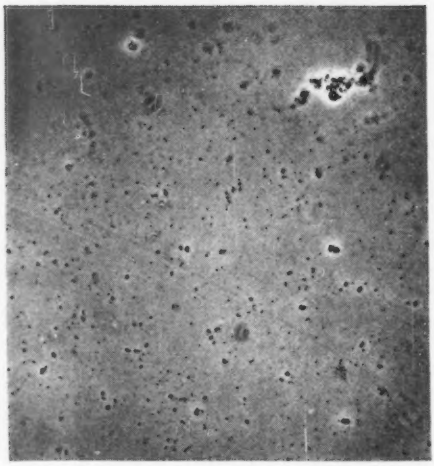

FIGURE 4. Dust, consisting mainly of quartz, collected on a Millipore type AA filter and cleared in a 1.507 liquid. Phase microscope × 210.

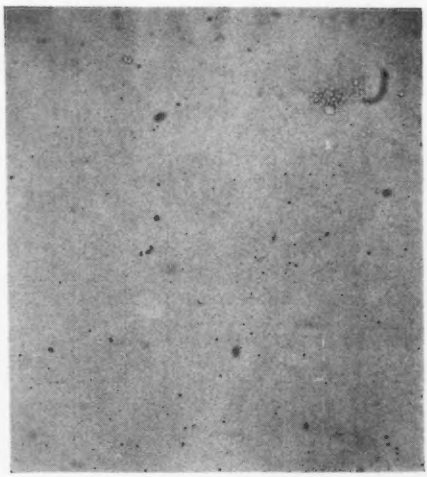

FIGURE 5. Exactly the same area as Figure 4. Cleared in a 1.507 liquid. Light-field microscope × 210.

working distance condenser having a working distance of 8–10mm. Use of this condenser was necessary due to the thickness of the counting chamber employed such as the Sedgwick-Rafter and Dunn cells. This condenser should also be used in the case of membrane filters used in conjunction with a hemacytometer as suggested in the first part of this paper. The underglass surface of the hemacytometer should have little or preferably no concavity. To prevent scratching of the glass in contact with the microscope stage some hemacytometers have their lower surface ground concave. A deep depression acts as a lens, making it impossible to center the image of the condenser with the phase annulus.

In the case of membrane filters placed dust side down on a microscope slide, it is possible to use either a long working distance condenser or the turret type Abbe Condenser of 1.25 N.A. Higher magnification objectives, such as the 43x, 4mm and 97x, 1.8mm, can also be used, allowing for increased resolution when necessary for small particulate material. A coverglass of approximately 0.18mm should be used with the 43x objective and a thinner coverglass with the 97x oil immersion objective.

Use of a cap analyzer over the eyepiece of the phase microscope is sometimes of value for the examination of birefringent dusts oriented such as to have either their ordinary ray or extraordinary ray very close to the index of the oil used for rendering the filter transparent. Rotation of this cap analyzer usually serves to increase the contrast of such particles. It is also possible to use a substage polarizer with the cap analyzer, a combination which is of value for determination as to whether the dust collected is isotropic or anisotropic. An interesting preparation to examine with these accessories is a mixture of opal silica

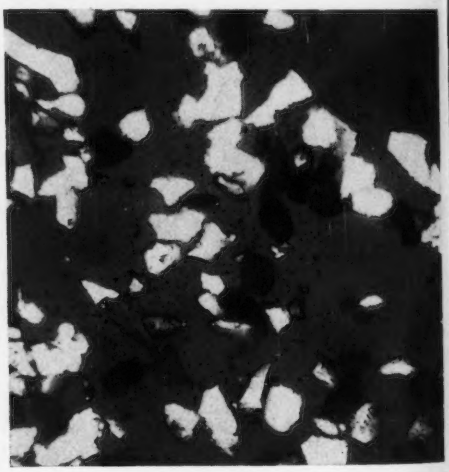

FIGURE 6. Mixture of opal silica and quartz silica on a Millipore type AA filter and cleared in 1.507 liquid. Phase microscope with polarizing accessories × 210.

and quartz silica on a Millipore filter type AA cleared with the 1.507 liquid. With the plane of vibration of the analyzer and polarizer at right angles to each other, opal silica, Figure 6 having no birefringence, will appear dark as compared to the bright particles of anisotropic quartz. A variation of the above is to combine a first order red retardation plate with the cap analyzer. With this combination, quartz will appear either blue or yellow dependent on its orientation unless at a point of extinction. Isotropic opal appears a pink to red color on the same colored background, at all points of orientation.

Summary

The use of phase microscopy is suggested for the examination of particulate material, such as calcined diatomaceous earth and quartz dusts, collected on membrane filters having refractive indices close to the index of the oil used for rendering the filter transparent. With this method of illumination, it was found that a clearing oil of approximately 1.507 in refractive index must be used in order to completely remove the texture of the filter.

Use of a cap analyzer and substage polarizer in combination with the phase microscope is of value for differentiation as to whether the dusts collected on the membrane filter are isotropic or anistropic.

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THE PAST PRESIDENTS OF AIHA were honored at the annual dinner meeting on April 29, 1959. This was also a celebration of the twentieth anniversary of AIHA. Nineteen of our Past Presidents are still living and seventeen were present at the banquet. J. J. Bloomfield and Dr. Philip Drinker were unable to attend because they were out of the country. Each of the Past Presidents was presented with a plaque expressing the Association's recognition and appreciation. Our immediate Past President parted with his plaque long enough that we might bring you this photograph of it.

An Investigation into the True Exposure of Arc Welders by Means of Simultaneous Sampling Procedures

WM. S. JOHNSON

Bethlehem Steel Company, Bethlehem, Pennsylvania

SAMPLING of welding fume for the purpose of determining a welder's exposure has always posed a question about the validity of the measured concentration of fume in a welder's breathing zone. This is so because the conventional method, e.g. the electrostatic precipitator, of necessity samples outside the welder's protective hood and, therefore, does not measure his exposure, but rather the concentration of metallurgical fume which surrounds him.

If one reflects on the actual situation that exists when a welder using conventional electrodes is at work, it becomes abundantly clear that his air supply (the air he actually inhales) contains much less welding fume than does the air at any point immediately outside his welding hood. The reason for this is presented in the following paragraph. It was the purpose of this investigation to determine the approximate magnitude of this relationship for welders in a large steel fabricating shop.

The air partially confined between the welding hood and the welder's head has a volume of about two liters, depending primarily on the size of the man's head. Consequently, when he first begins welding, or after having placed a new electrode in the holder, he confines this amount of relatively clean air in his immediate breathing zone by flipping the hood into the welding position as he strikes the arc. At the first inhalation he takes about 0.7 liter of this relatively clean air into his respiratory tract. That amount of original air between the welder's head and the hood is replaced by the relatively contaminated air around the periphery of the hood; inward flow taking place at all points. Upon exhaling, a similar quantity of relatively fume-free air is blown back into the confined space under the hood thereby, displacing an equal amount of the air between the hood and the wearer's head. This pumping action continues until the electrode has been consumed—a matter of approximately two minutes, as a rule.

Because of the partial confinement of this originally rather clean air, and because the amount being exchanged back and forth by this reciprocating action is only about ⅓ of the total amount between the hood and the welder's head, the fume concentration of the air under the hood does not approach that in the air around the hood until the time the electrode is consumed and welding ceases. At this time the whole cycle is repeated, beginning again with relatively clean air under the hood. Admittedly there are many variable factors at play which govern the rate at which the air beneath the hood becomes contaminated. If this were not so, one could calculate rather accurately the concentration under the hood at any moment in terms of that at the periphery where the interchange takes place. However, to anyone who has observed welders at work and who has developed an accurate mental picture of the interchange between the air inside the hood and that outside as the wearer breathes, it will be apparent that the average welding fume concentration inhaled by the welder probably is less than half that prevailing directly in front of the hood in the area where samples normally are taken for measuring the welder's exposure.

For the purpose of the study a conventional welder's hood was fitted with a one inch diameter aluminum filter holder, which placed the filter immediately in front of the nose and mouth of the wearer of the hood, as shown in Figure 1. The filter medium used was glass fibre filter web cut into one-inch diameter circles. To this a midget impinger pump was connected and sampling was done at the rate of five to six liters per minute. The steady withdrawal of air from the breathing space by means of the midget impinger pump increased the rate at which the air beneath the hood became contaminated with welding fume, so that the filter concentrations reported here are even greater than would normally be present.

Samples were collected in two welding shops where steel castings and weldments in sizes ranging from ⅛ ton to 10 tons were repaired and fabricated. No sample was taken under simulated conditions; all samples represent usual environment and situations found in welding shops. The

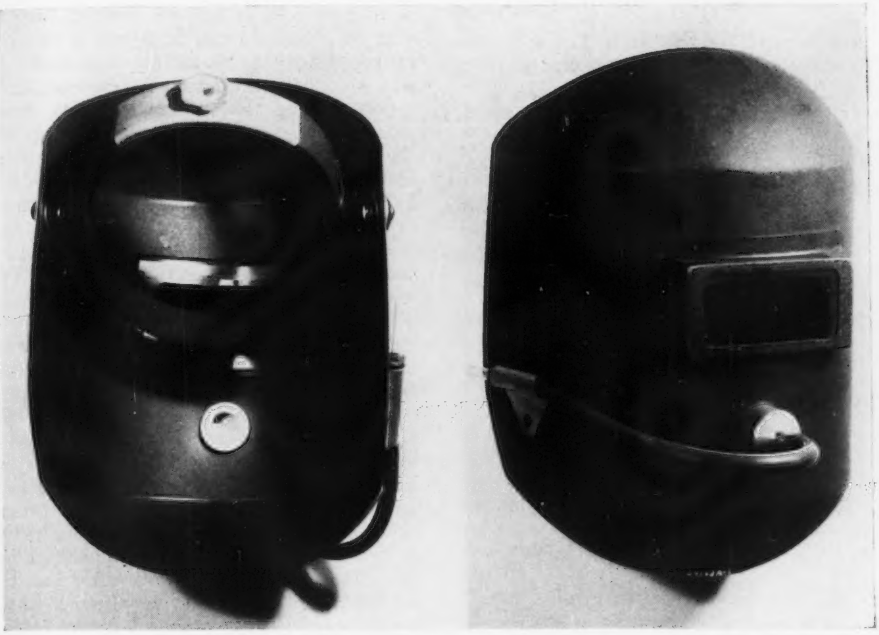

FIGURE 1. Rear view (left) and front view (right) of welding hood fitted with filter holder for sampling air inside the helmet during use.

actual sampling covered a period of four months (July–October) and represents conditions found during summer and early autumn months when natural ventilation would be expected to be at maximum.

Simultaneous sampling of fume was done by means of the filter heretofore described and an electrostatic precipitator. The precipitator was held about four inches forward of the welder's ear and within one inch of the welding helmet at the same level as the filter holder. Regardless of the direction of the moving air the precipitator samples were taken on both sides of the welding helmet, alternating each time a rod was consumed, thereby insuring as nearly as possible representative sampling of the welder's immediate environment outside the hood.

To test the inherent sampling efficiency of the filter, compared with the precipitator, three samples were taken in a plume of welding fume with the filter holder and the precipitator tube side by side. An attempt was made to sample light, medium and heavy concentrations. Table I shows the findings.

Samples of the fume were analyzed for iron oxide using a modification of the o-phenanthroline method of Pepi.[2]

It was impracticable to measure accurately the concentration represented by the filter samples, because of the change in air flow through the filter as the filter cake increases in density. Sampling was done at a constant pressure differential, but flow had to be determined by taking an average of the initial and final flow rates for each filter used. While such appears unlikely, it may be that this measuring was responsible for the difference between the measured concentrations by the two sampling methods.

TABLE I

Relative Concentrations Found by Two Sampling Methods

(Simultaneous samples of same air source.)

Method	Condition	Concentration Fe_2O_3 mg/m³	Ratio of ESP concentration to filter concentration
Filter	Light	4.02	1.10
ESP	"	4.41	
Filter	Medium	9.30	1.08
ESP	"	10.00	
Filter	Heavy	30.50	1.10
ESP	"	33.50	

TABLE II

Comparison of Welding Fume Concentrations Inside and Outside the Welder's Hood

(Expressed as Mg Fe_2O_3/M^3 of air)

ESP samples outside of helmet	Filter samples inside of helmet	Ratio of ESP to filter concentrations
1.54	1.03	1.50
1.99	1.13	1.76
2.04	0.99	2.10
5.70	2.51	2.27
11.7	11.4	1.03
25.5	8.33	3.06
29.7	18.4	1.61
10.9	2.60	4.19
6.24	2.15	2.94
4.43	2.03	2.18
8.60	2.93	2.93
37.6	8.47	4.45
11.3	3.35	3.38
3.29	1.31	2.51
4.33	. 1.88	2.30
61.2	23.8	2.57
69.7	27.3	2.55
74.5	25.1	2.98
5.0	1.28	4.53
4.54	2.70	1.67
13.50	4.07	3.32
9.20	3.04	3.03
15.5	4.42	3.52
8.35	4.39	1.90
20.8	4.71	4.42
4.36	2.32	1.88
7.96	5.16	1.54
5.46	1.76	3.10
7.44	3.80	1.96
34.5	4.60	7.51
22.7	5.07	4.49
48.9	6.88	7.11
70.2	9.31	7.55
17.9	3.43	5.23
20.0	3.24	6.25
6.74	3.53	1.90

Reference to Table II will show that the concentration of fume collected by the electrostatic precipitator is about 3.5 times greater than that collected by the filter. The range of ratios extends from 1.03 to 7.55, the average being 3.5. Samples showing ratios of 6.0 or more can be explained by the conditions under which the samples were taken. In all of these cases the air motion around the welder tended to channel most of the fume into the area being sampled by the precipitator. Mitigating circumstances can also explain the ratios below 2.0 on the same basis, i.e. air motion tended to reduce the amount of fume present in the sampling area to a minimum. It must be emphasized that this study was made under normal operating conditions in the welding shop and no attempt was made in either direction to influence the collection of fume.

The results from this study show that the concentration of welding fume immediately outside of a welder's hood in the area normally sampled for measuring a welder's exposure is considerably higher than that in the air actually breathed by the welder. This investigation has been preliminary in character and purports to show nothing more than the marked difference between fume concentrations outside and inside the welder's hood. McDaniel[2] in an unpublished work found the same conditions, but aside from that the literature is quite devoid of work on this specific subject. The methods described here are not ideal for the purpose. The use of a mechanical filter respirator worn by the welder, the total volume of air "sampled" to be determined by spirometry, would be better in that the rate of air exchange between the "confinded" space under the hood and the outside would not be altered. In any event, it is believed that this problem deserves further investigation by other means.

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The Pulmonary Response to Blast Furnace Stack Dust*

PAUL GROSS, M.D., MARIAN L. WESTRICK, Ph.D., and JAMES M. McNERNEY, M.P.H.

Industrial Hygiene Foundation, Pittsburgh, Pennsylvania

INTRAPULMONARY IRON deposits are not known to produce clinical disease even though such deposits may produce roentgenologic opacities which, to the inexperienced eye, may suggest an alarming degree of disabling disease.[1] In this laboratory, iron (as ferric oxide) has behaved like an inert dust and has caused no greater connective tissue response in the lungs of experimental animals than has carbon or clay (kaolinite).[2] As a matter of fact, iron was found to be capable of inhibiting the fibrogenic action of silica in the lungs of animals as early as 1932.[3] This ability of iron to inhibit or modify silicosis has been confirmed by a number of other observers.[4, 5, 6]

The solid constituents of stack effluents from blast furnaces may contain a considerable amount of iron together with siliceous and other components. In spite of the most modern equipment, a very small percentage of such stack effluents may defy entrapment and escape into the atmosphere, thus constituting an air pollutant capable of being inhaled by people in the vicinity of such plants. What effect the solid constituents of stack effluents from blast furnaces would have on the lungs of experimental animals therefore has some practical as well as scientific interest.

The purpose of this communication is to report the results of an investigation in which the solid constituents of stack effluents from a blast furnace were injected into the lungs of rats as a single moderately large dose and also as multiple large doses repeated as many as eight times over a period of six months.

Methods and Materials

In order to give added meaning to this investigation, the pulmonary injections with blast furnace stack dust were paralleled in other animals by similar injections of kaolin dust and quartz dust respectively. The latter two dusts were to furnish bench marks to which the effects of the stack dust were to be compared.

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The blast furnace stack effluent dust used for the injections was considered representative and consisted of a dark red-brown powder which had the composition given in Table I. A quantity of this dust was reduced in particle size to an average of 1.7 μ. (Surface mean diameter = $\Sigma nd^3/\Sigma nd^2$.) Another portion of the dust was suspended in water and allowed to settle for five minutes, and the top portion was poured off for subsequent use. Aqueous dust suspensions were standardized to contain 25 and 30 mg dust per ml, respectively. The average particle size in these suspensions was 5.8 μ.

Quartz dust suspensions were prepared from ground flint in identical fashion and kaolin dust suspensions were also similarly prepared. The latter were 98% pure kaolinite and contained less than 0.5% crystalline silica. The average particle sizes of the experimental dusts are given in Table II and their particle size distribution, in Table III.

A total of 180 rats were used. These were divided into two series. One series consisted of three groups of 30 rats each which were given a single intratracheal injection of 30 mg of the respective dusts with the smaller average particle size. The second series, also consisting of three groups of 30 rats each, were given multiple injections of the respective dusts, but of the larger particle size. The first injection of the second series consisted of 30 mg, but subsequent injections were 25 mg. The first two injections were two weeks apart. A total of 230 mg of dust was thereby administered. At the end of this time it was thought that the few rats which had survived in the quartz group could not withstand further dosing. It was therefore decided to stop all injections.

The intratracheal injections of kaolin and quartz dust with the very small average particle size were associated with a high immediate mortality.[7] It became necessary to administer the fine quartz dust in two doses of 15 mg each, five days apart in order to obtain 30 survivors. In the case of the kaolin dust, a sufficiently large number of rats were injected until 30 survivors were obtained (the mortality rate was approximately

TABLE I

Chemical Analysis of Blast Furnace Stack Dust

Total Fe (as Fe_2O_3)	76.03%
Si (as SiO_2)	8.72
Carbon	7.07
Al_2O_3	3.33
CaO	1.52
MnO	0.58
H_2O	0.51
K_2O	0.48
MgO	0.40
P_2O_5	0.27
S	0.22
CuO	0.01
Li_2O	0.00
As, Pb	not detected
Total	99.14%

TABLE II

Average Particle Size of Experimental Dusts*

Dust suspension	Average particle size† in microns		
	Stack effluent	Silica	Kaolin
Small particle size	1.7	1.2	0.7
Large particle size	5.8	2.4	2.3

* Determined by optical microscopy at 970×, oil immersion.
† Surface mean diameter = $\Sigma nd^3/\Sigma nd^2$.

50%). The injections were accomplished by means of a self-retaining, illuminated laryngoscopic speculum[5] under light ether anesthesia.

All animals of the first series were killed with ether 18 months after the initiation of the study, and those of the second series were sacrificed 14 months after the initial injection and 7½ months after the last. A few rats from each group were killed at intervals for the purpose of sampling but many of the animals died of intercurrent disease. All animals were autopsied, the lungs inflated with formalin and paraffin sections were prepared in the usual manner. In addition to the usual staining procedures, incineration studies were also performed.

Results

General

Because of endemic pulmonary disease, there was an appreciable mortality rate from spontaneously arising pneumonia and complications such as empyema. This was undoubtedly aggravated by the intratracheal manipulations incident to the dust injections. In the case of the single (with silica, two-fold) injections, the mortality from such spontaneous infections averaged 33%. With the multiple intratracheal injections, the mortality was appreciably higher. The cumulative mortality rates of the different groups are listed in Table IV.

The greatly higher mortality rate of the cumulative quartz dust injections was caused by massive silicotic involvement of the lung tissue that left an amount of functioning parenchyma insufficient to support life.

Stack Dust (single 30 mg dose)

Grossly, the only deviation from normal was a mottled black pigmentation of the pleura and the cut surface.

TABLE III

Particle Size Distribution of Experimental Dusts

Particle size (microns)	Stack effluent		Silica		Kaolin	
	% by No.	% by Mass	% by No.	% by Mass	% by No.	% by Mass
Large particle size suspensions						
<0.4	5.5	0.0	7.0	0.0	6.5	0.0
0.4	8.0	0.0	12.0	0.2	22.0	0.5
0.6	10.5	0.0	15.0	0.7	29.5	2.3
0.9	14.0	0.3	24.5	3.7	22.5	5.8
1.3	16.0	0.9	17.5	7.9	9.5	7.4
1.8	13.5	1.9	14.5	17.4	4.5	9.4
2.5	16.5	6.3	5.5	17.7	2.5	14.0
3.5	8.5	8.9	3.0	26.6	2.5	38.3
5.0	2.5	7.6	1.0	25.8	0.5	22.3
7.0	3.0	25.2	—	—	—	—
10.0	2.0	48.9	—	—	—	—
Small particle size suspensions						
<0.4	38.8	0.3	64.8	1.2	52.0	3.5
0.4	17.6	0.8	10.4	1.6	33.2	18.1
0.6	12.2	1.9	6.0	3.1	9.6	17.6
0.9	11.6	6.3	8.6	14.8	3.4	21.1
1.3	11.0	17.8	7.6	39.0	1.6	29.8
1.8	6.2	26.7	2.4	33.0	0.2	9.9
2.5	1.8	20.8	0.2	7.3	—	—
3.5	0.8	25.4	—	—	—	—

TABLE IV

Cumulative Mortality (Spontaneous Deaths) following Multiple Intratracheal Dust Injections

Dust	Months after first injection			
	4	6	9	14
Stack	27%	27%	40%	50%
Quartz	63%	80%	87%	90%
Kaolin	43%	53%	53%	57%

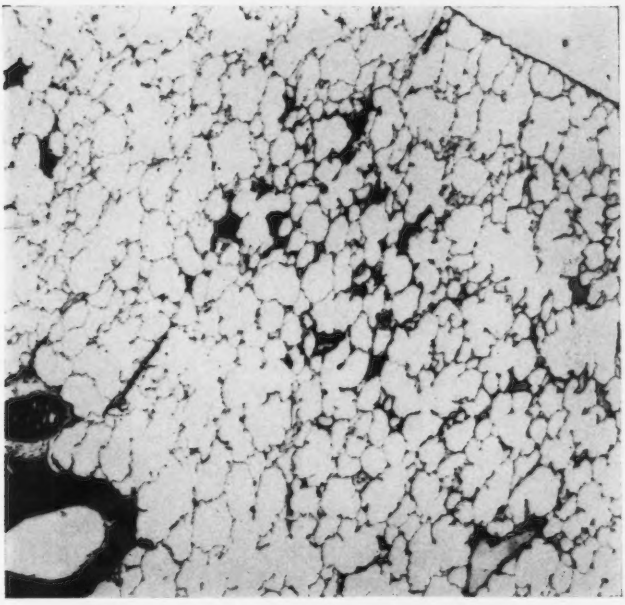

FIGURE 1. Rat 3437 given a single intratracheal injection of 30 mg blast furnace stack dust and killed 12 months later. This field is representative of the findings in most animals of this group. Relatively few dust foci are present. These consist of dust particles densely aggregated within macrophages which, in turn, are so closely packed as to form solid masses within air spaces. Very little interstitial dust is present. Alveolar walls are normal.

FIGURE 2. Same rat as in Figure 1. This field is from a different lobe in which chronic purulent bronchitis is present. As a result there has been bronchial obstruction, atelectasis, and interference with alveolar clearance. There appear to be many more dust foci than are seen in Figure 1. Also, because of peribronchial inflammation some dust foci are broken up into loosely aggregated pigmented macrophages.

FIGURE 3. Same field as in Figure 2. This is a composite photograph made by the superimposition of the dark-field negative of the acid-washed ash upon the light field negative of Figure 2. It is interesting to note that some of the dust foci contain much acid-insoluble ash (silicates, silica) whereas others seem to contain little or no ash. In contrast, most of the macrophages contain much ash. The bronchus in the lower left shows much finely divided ash in the purulent exudate even though very little iron is present.

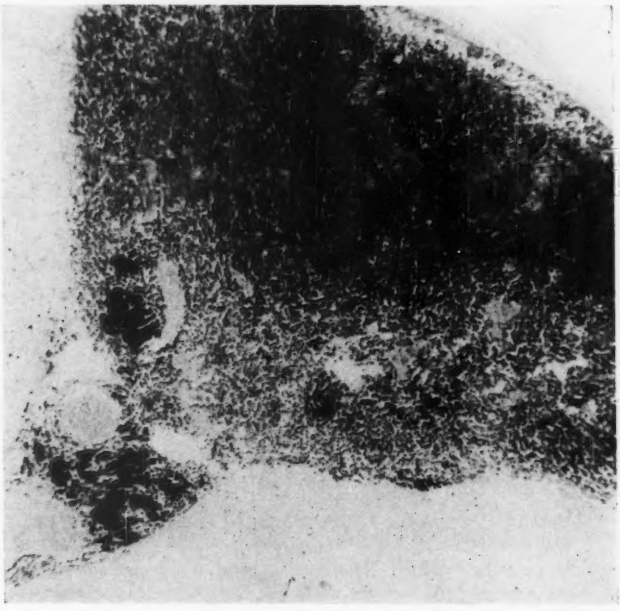

FIGURE 4. Same animal as in Figure 1. Paratracheal lymph node showing minimal dust deposition. The dust is inert inasmuch as there is no discernible reaction to its presence.

Microscopically, the main lesion was likewise solely one of pigment deposition. There were no significant differences in the appearance of the lungs examined at different time periods following the intratracheal injection. The distribution of the dust lacked uniformity. In some animals, the dust was confined largely to one lung, and in others one or several lobes were found practically devoid of dust. Even within the lobes, the distribution of the dust was irregular and did not follow any pattern.

The dust formed dense black pigment masses which filled clusters of air spaces. The pigment was so densely aggregated that macrophages could not be identified. However, there were many areas, particularly near foci of inflammation, where disintegration of the alveolar pigment masses seemed to be in progress. Here it could be established that the dust was intracellular and that the pigment masses were composed largely of densely packed, dust-filled macrophages. Interstitial deposition of dust was scanty and could be demonstrated with certainty only in the loose periadventitial stroma of some vessels and bronchi where it was likewise intracellular. In regions of frank pneumonia, it was uncommon to encounter more than an occasional pigmented macrophage. Many of the animals suffered from chronic bronchitis endemic to rats. As a result, one or several lobes of these animals frequently was atelectatic due to bronchial obstruction. Such atelectatic lobes were commonly heavily burdened with dense dust deposits diffusely distributed.

No connective tissue fibers could be demonstrated in connection with the dust deposits but some alveolar walls were thickened by swelling and proliferation of the alveolar lining cells.

In harmony with the paucity of interstitially situated dust, little or no dust deposition could be found in the pulmonary lymph nodes. As a matter of fact, more than one-half of the nodes encountered in the sections failed to demonstrate any pigment whatsoever. However, when dust was present in the lymph nodes, it appeared to be inert because no reaction to its presence was demonstrable.

Kaolin Dust (single 30 mg dose)

Similar to the above stack dust animals, the only evidence of kaolin dust injection visible to the naked eye was a slight mottling by opaque gray pigment upon the pleura and upon the cut surface.

Microscopically, the kaolin was disposed in well-circumscribed, frequently angulated foci composed of closely packed, large, pale macrophages filled with fine, nearly colorless particles. Very fine collagen fibers could occasionally be found coursing among these dust cells. Similar and thicker collagen fibers were more often found on the periphery of the dust depots. The presence of collagen fibers within and about the kaolin dust foci justifies their classification as interstitial. In regions of edema and pneumonia, the kaolin dust depots were more difficult to find and often the only indication of dust was the presence of scattered dust-filled macrophages.

In keeping with the predominantly interstitial position of the dust depots, all nodes found in the sections contained kaolin deposits. Such deposits consisted of rounded foci of large, reticulum-like cells filled with dust granules. No collagen fibers were seen in association with these dust cells.

Quartz Dust (single or divided 30 mg dose)

Grossly, the animals in this group showed scattered silicotic nodules upon the pleural and cut surfaces. In some animals, an atelectatic lobe was studded with such nodules. The tracheal lymph nodes were usually enlarged and hard.

Microscopically, the sections of the lungs were characteristic of silicosis as usually encountered in this animal. The nodules showed varying degrees of collagenization. In atelectatic lobes, the nodules tended to be confluent. Also, the collagenization tended to be more extensive. The tracheal lymph nodes were involved by a combination of collagenization with masses of reticulum cells that, in large part, replaced lymphocytic tissue.

Stack Dust (multiple doses)

Grossly, the results differed from those of single doses only in the intensity of the pigmentation.

Microscopically, the sections were characterized by larger and more numerous foci of pigmentation than was the case in animals given the single dose. The nature of the dust deposits was identical. Relatively little pigment was found interstitially and the lymph nodes also contained little dust. No fibrous tissue response was found in the lungs or the lymph nodes secondary to the rust deposits. In regions of the heaviest dust deposits, considerable swelling and proliferation of alveolar cells gave the alveolar walls a thickened appearance. Between the involved foci, the lung tissue was essentially normal. In pneumonic regions, the dust depots showed evidence of disintegration as indicated by the large number of discrete, dust-filled macrophages and the reduction in the number of dust foci.

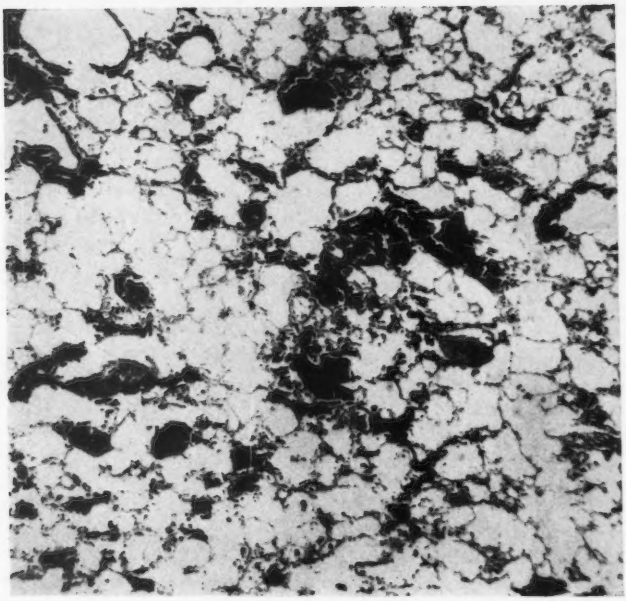

FIGURE 5. Rat 3639 killed 14 months after the first intratracheal injection. It was given injections for a total of 230 mg. Dust foci are intraalveolar, larger, and more numerous. Little dust is found interstitially. There is no connective tissue reaction. Alveolar walls are normal. Many loose macrophages are present.

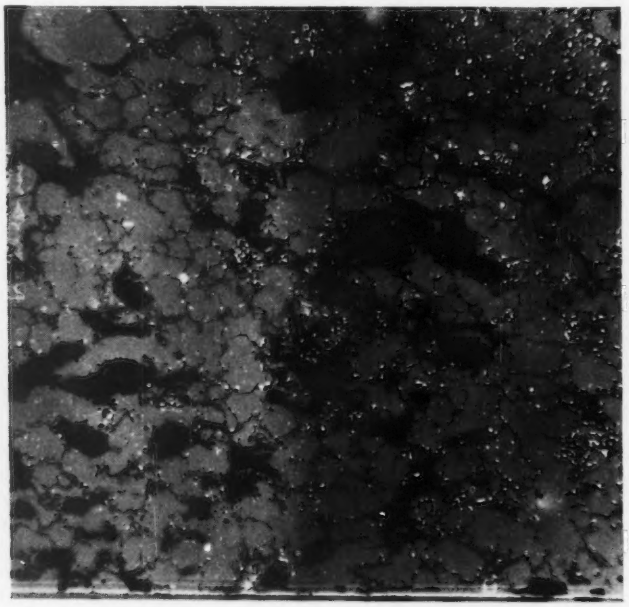

FIGURE 6. Same field as in Figure 5. This is a composite photograph and shows that the large dust foci contain virtually no acid-insoluble ash whereas the loose alveolar macrophages contain an abundance of such ash.

Kaolin Dust (multiple doses)

The gross apperance of the lungs of this group was similar to that of the group given the single dose of the same dust except for a more pronounced pigmentation.

The microscopic features resembled those of the group given but a single dose of kaolin dust only in the paucity of the connective tissue reaction. The dust foci tended to become confluent or interconnected and were more numerous and larger than those encountered in the single-dose group. Also, instead of being composed of larger, pale macrophages, many of the dust foci were composed mostly of crowded foreign-body giant cells and large intracellular, spiculated crystals. There was focal cellular thickening of alveolar walls. Compared to the lesions caused by stack dust, the multiple kaolin injections seriously impaired a much greater amount of alveolar tissue.

The tracheal lymph nodes contained masses of crowded reticulum cells filled with fine dust granules similar to the nodes associated with the single-dose animals. No collagen production could be demonstrated in these lymph nodes.

Quartz Dust (multiple doses)

As indicated in Table IV, most of these animals died before the experiment was terminated. The lungs of these rats were nodularly consolidated and hard. The cut surfaces resembled cartilage and showed very little spongy lung tissue. The impression was gained that many of these animals died, not so much of an incidental pneumonia as of respiratory inadequacy. The lymph nodes draining these lungs were uniformly enlarged and hard.

Microscopically, the silicotic nodules were of varying degrees of maturity with extensive collagenization and confluence. In addition, the air spaces between the nodules tended to be filled with desquamated alveolar macrophages, many of which were lipidic.

The tracheal lymph nodes were the seat of extensive fibrosis and masses of large reticulum cells filled with quartz dust which had replaced lymphocytic tissue.

Incineration Studies

In the single dose series of animals the kaolin ash pattern gave the usual solid appearance of the individual dust foci in both lung and lymph node whereas the silica ash pattern was flocculent. The ash pattern of the stack dust, on the other hand, was irregular. Many of the dust foci contained no acid-insoluble ash while others contained abundant densely aggregated mineral. In regions of inflammation, where dust foci had been broken up into their component macrophages, most of the macrophages contained a large amount of acid-insoluble ash in addition to their high iron content. The distribution of the ash within the stack dust foci in the lymph nodes was similarly irregular.

In the multiple dose series the stack dust foci in the lungs were mostly devoid of acid-insoluble ash although nearby loose, pigmented alveolar macrophages generally contained appreciable ash. Very little ash could likewise be demonstrated in the stack dust foci of lymph nodes. There was a considerable reduction (compared to the single-dose animals) in the mineral content of silicotic nodules and of the kaolin foci.

Discussion

There are two highly significant features of the pulmonary reaction to blast furnace stack dust. The first is that in the rat there is no discernible connective tissue reaction to the presence of the dust in either lung tissue or lymph node. This would seem to classify this dust as even more inert than kaolinite in which a minimal connective tissue reaction is usually demonstrable. The second feature is the storage of dust almost exclusively in the air spaces and its extremely limited ability to penetrate the lung interstitium.

According to a recently suggested modified concept of pneumoconiosis, dust may penetrate the alveolar membrane only as naked particles.[8] Dust which has been ingested by phagocytes is rendered harmless and is incapable of penetrating into the lung interstitium as long as it remains inside of the cell. Since stack dust is readily phagocytosed and very little, if any, was found in extracellular position, these facts offer a reasonable explanation for the limited ability of the stack dust to penetrate into the lung stroma and also for the extremely small amount of dust found in the tracheal lymph nodes.

The relatively normal state of alveolar walls in spite of the tremendous overload of stack dust introduced into the lungs by multiple injections underscores the biologic inertness of this dust. The inability of significant amounts of stack effluent dust to penetrate either the lung stroma or to reach the draining lymph nodes even under the conditions of this large overload, adds further emphasis to this conclusion.

The difference in the character of the response to the kaolin dust given as a single injection and that resulting from the multiple injections can be ascribed mostly to the larger particle size associated with the multiple injections.

The greater amount of lung tissue rendered

functionless by the multiple kaolin injections than by the multiple stack dust injections can be attributed, in part, to the inability of the alveolar clearance mechanism to rid the air spaces of the large kaolinite crystals. The stack dust, on the other hand, was much more readily cleared thereby allowing less of the dust to block the air spaces. Another reason for a greater incapacity caused by kaolin dust than by stack dust resides in the tendency of kaolin-filled cells to be anchored to the alveolar walls by a scanty and delicate connective tissue stroma which further hinders its removal from the lung. No such impediments to the removal of stack dust deposits have been noted.

The irregular pattern of the acid-insoluble ash which is seen in the single-dose animals injected with stack dust indicates that dissociation of the dust components had occurred. That is, although the iron was originally associated with silicates and some of the dust foci gave evidence of such association, other dust foci were devoid of mineral dust. Of further interest is the fact that in the vicinity of ash-free dust foci loose alveolar macrophages do contain appreciable ash as well as iron. How and why such dissociation occurs is probably related to the problem of the mechanics of alveolar clearance about which very little is known.

This dissociation of mineral from iron is nearly complete in the multiple-injection group where it was undoubtedly aided by recurrent episodes of edema caused by each of the multiple intratracheal injections. The recurrent episodes of edema probably also explain the reduction in mineral content of both, silicotic nodules and kaolin foci in this series as compared with the mineral content of these structures in the single-injection series.

Summary and Conclusions

A representative blast furnace stack dust was injected intratracheally into rats as a single dose and also as multiple doses.

The pulmonary response to these injections was compared to the reactions obtained in comparable groups of rats similarly injected with kaolin and quartz dust respectively.

The blast furnace stack dust provoked no discernible connective tissue reaction in either lung or lymph node and demonstrated an extremely limited ability to penetrate the lung interstitium; this, in spite of very heavy dosage. The blast furnace stack dust thereby proved to be even more inert than kaolinite.

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Industrial Hygiene Experiences with Toluene Diisocyanate

HERBERT T. WALWORTH and WARREN E. VIRCHOW*

Lumbermens Mutual Casualty Company, Chicago

BASIC RESEARCH in polyurethane chemistry was started in the 1930's but it was not until recent years that consumer products were manufactured on a large scale. Perhaps the greatest industrial activity in this country has been the manufacture of flexible foams. These foams are used as substitutes for foam rubber and a host of new products which are finding wide acceptance.

This paper concerns the results of observations and field studies involving two principal industrial operations; namely,

(1) The continuous production of a foam slab approximately 48 inches wide and 12 to 16 inches thick.

(2) The production of molded products of varying sizes and shapes.

The principal components of polyurethane chemicals are isocyanates and polyester or polyether resins. When these chemicals are brought together in the presence of water and a catalyst under proper conditions, linkages are formed between the polyester or polyether and isocyanate. In the formation of certain of these chemical linkages, carbon dioxide is produced. This gas causes the resin to rise in a manner similar to the rising of bread dough.

The basic ingredients for flexible foam production include:

(1) polyester or polyether resins,

(2) combinations of toluene 2,4-diisocyanate and toluene 2,6-diisocyanate (TDI),

(3) catalysts,

(4) dyes,

(5) emulsifiers,

(6) water.

There are hundreds of combinations of these materials which can be mixed to produce foams of varying densities and degrees of rigidity.

Toxicity of TDI

According to Zapp[1], toluene diisocyanate has a low acute toxicity, but it is irritating to the skin, eyes and respiratory tract. Contact with the liquid or vapor may cause allergic skin sensitization, and inhalation of the vapor may cause allergic asthmatic reactions. The polymers formed with this chemical are physiologically inert.

General Factors Affecting Exposures to TDI

When the polyurethane chemicals are brought together, foaming starts almost immediately, but thorough mixing is necessary to assure a uniform product. Depending upon a variety of factors including temperature and percentage of catalyst, the foaming is completed in a period ranging from a few minutes to perhaps an hour or more.

The chemical reaction in this process is exothermic. This can be of considerable significance in the rate and volume of TDI vapor production, and thus the hygienic exposures in connection with certain processes. As the chemical reaction progresses, the excess carbon dioxide liberated carries with it a certain amount of TDI vapor which can cause workroom contamination.

TDI has a low vapor pressure. Its boiling point of 247°C is high when compared with other common industrial liquids having hygienic significance. These properties tend to provide inherent vapor control. However, during the foaming process the temperature rises which tends to increase the amount of TDI liberated into the atmosphere of the workroom.

Polyether resins produce a sealed cell structure in the foam which shrinks upon cooling. These cells are broken by rolling with further liberation of carbon dioxide gas and TDI vapor.

Operations involving TDI were started early in 1955 in the plant studied. Production reached a maximum during the late summer and early fall of that year. However, it was not until the beginning of cool weather that general complaints and absenteeism became a problem.

UBK Machine Operation

The UBK machine (Figure 1) is equipment which is designed to produce wide and thick slabs of polyurethane foam. The finished foam is cut and sliced into products of various sizes and shapes for numerous applications. This machine

* Present affiliation: American Cyanamid Company, Stamford, Connecticut.

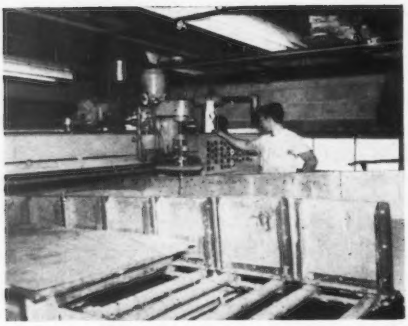

FIGURE 1. General view of mixing end of UBK machine.

operates with a minimum of operator attention. It is approximately five feet wide and forty feet long. It is enclosed except at the ends and the enclosure is provided with exhaust ventilation.

The polyurethane chemicals are pumped to a nozzle where they are mixed and delivered to a conveyer moving slowly through the machine enclosure. The nozzle travels back and forth across the conveyer, depositing a layer of liquid approximately three-eighths inch thick. Depending upon such factors as nozzle adjustment, conveyer speed and temperature, the liquid foams to a height of 12 to 16 inches (Figure 2).

When the nozzle is put into operation and at the end of the mixing process, a certain amount of unreacted polyurethane mixture accumulates. The amount of accumulation depends upon the difficulties encountered in nozzle adjustment and upon other operational factors. Originally it was common practice to allow these scraps to accumulate in the open, with a resultant increase in TDI exposures. Later these scraps were placed routinely in a ventilated enclosure.

As the foam slab emerges from the UBK machine, it is usually cut into pieces about six feet long. The cakes may be placed in heated curing ovens or stacked in a storage area to await further processing.

The following operating practices in UBK machine operation were found to affect the TDI exposures materially:

1. Manipulation of the nozzle by the operator.
2. Trial runs producing an abnormal amount of freshly mixed polyurethane chemicals which, if left in the open, contributed to workroom contamination.
3. Spillages of TDI liquid caused by transfer, handling, equipment leaks, or maintenance operations.
4. The continued evolution of TDI vapor for some time after the slabs are removed from the UBK machine. Cake temperatures may reach 150° to 160°C, and in some cases the slabs emerging from the machine are as little as 40% cured.

Production of Molded Products

During the period of this study, the production operations involved in the manufacture of molded foam products underwent numerous technological changes. Some of these changes were favorable to TDI vapor control; others were production improvements only.

Initially, the operations were almost entirely

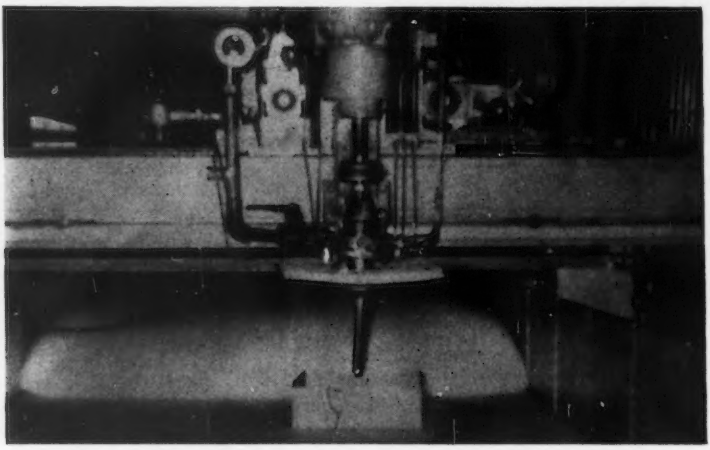

FIGURE 2. Close up of delivery nozzle of UBK Machine.

manual. The resin, water, emulsifier, dye and catalyst were premixed and weighed into a paper cup. The TDI was added through a foot-operated measuring valve and the contents were mixed by holding the cup under a stirring device for about 20 seconds. The paper cup was then inverted and squeezed by hand over an open metal mold (Figure 3). The mold was closed and manually pushed onto a conveyer leading to a heated tunnel. Workers at the exit end of the tunnel opened the mold, removed the piece, and transferred the mold to a return conveyer.

The operation involving the measuring of TDI liquid and the mixing of the chemicals was partially enclosed and the enclosure was provided with mechanical exhaust ventilation[3] (Figure 4).

Later, the process was changed so that the resin, TDI, water, emulsifier and dye were mixed under controlled conditions to form a prepolymer. This was then weighed into paper cups and the catalyst was added just prior to mixing and addition to the mold. This was a decided improvement over the previous method, but it remained a manual operation offering a number of possibilities for contamination with TDI vapor. Since the period of this study there has been a gradual conversion to automatic measuring.

The following operations contributed to TDI vapor exposures in molded product operations:

(1) TDI liquid spillages at the mixing station.

FIGURE 3. Hand-injection of TDI mixture into molds by means of a paper cup.

(2) Filling the molds with mixed polyurethane chemicals.

(3) Closing the molds and passing them to the heated tunnel.

(4) Removing the product from the molds.

(5) Exposing scrap paper cups to the workroom allowing escape of TDI vapors.

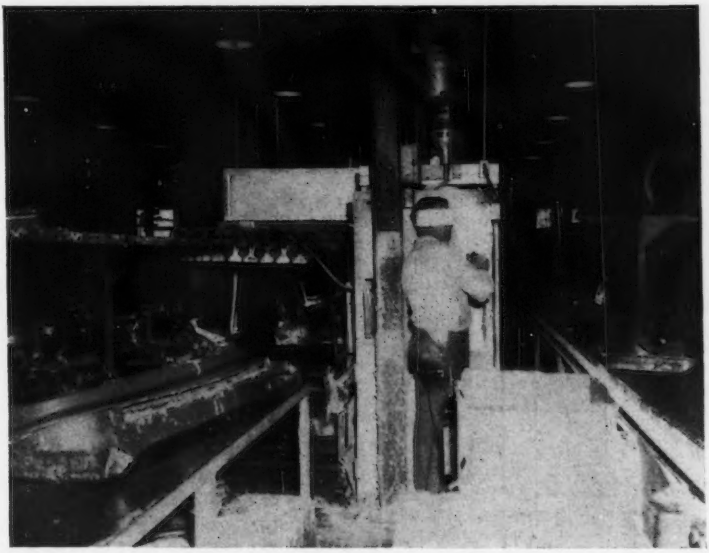

FIGURE 4. Measuring TDI liquid and mixing polyurethane chemicals in a ventilated enclosure. Entrance conveyer on left. Return conveyer on right.

(6) Accumulation of spillages of TDI liquid, prepolymer, and polymer.

Study Procedures

Early in October, 1955, a case of asthma developed in a worker in the plant in question. The condition was tentatively associated with the polyurethane operations by the plant physician.

TABLE IA
Summary of TDI Vapor Concentrations, 1955–57
(parts per million by volume in air)

Date	Pouring area			Extra crash pad			Visor table			Crash pad table and preform area		
	Ave.	Min.	Max.	Ave.	Min.	Max.	Ave.	Min.	Max.	Ave.	Min.	Max.
Oct. '55	0.21	0.07	0.38	0.16	0.07	0.35	0.04	0.02	0.08			
Jan. '56	0.03	0.01	0.10	0.01	0.01	0.02	0.02	0.02	0.03	0.02	0.01	0.03
Feb.	0.13	0.02	0.68	0.03	0.02	0.05	0.44	0.02	0.82	0.02	0.01	0.04
March	0.22	0.01	2.2	0.02	0.01	0.03	0.34	0.05	1.2	0.01	0.01	0.02
April	0.03	0.01	0.09	0.01	0.00	0.01	0.25	0.00	0.59	0.01	0.01	0.02
May	0.02	0.00	0.06	0.01	0.00	0.01	0.02	0.00	0.05	0.01	0.00	0.02
June	0.03	0.01	0.07	0.01	0.00	0.02	0.01	0.00	0.05	0.01	0.00	0.01
July	0.02	0.00	0.05	0.00	0.00	0.00	0.01	0.00	0.01	0.00	0.00	0.00
Aug.	0.01	0.00	0.02	0.00	0.00	0.00	0.01	0.00	0.01	0.01	0.00	0.01
Sept.	0.03	0.03	0.03	0.02	0.00	0.03						
Oct.	0.01	0.01	0.02							0.03	0.00	0.07
Nov.	0.02	0.02	0.02									
Dec.	0.03	0.02	0.03							0.03	0.02	0.03
Jan. '57	0.05	0.01	0.10	0.05	0.03	0.08						
May	0.05	0.05	0.05									
Dec.				0.00	0.00	0.00						

TABLE IB
Summary of TDI Vapor Concentrations, 1955–57—continued
(parts per million by volume in air)

Date	Arm rest table and area			UBK machine exit and curing area			Prepolymer mix house			Miscellanous areas		
	Ave.	Min.	Max.	Ave.	Min.	Max.	Ave.	Min.	Max.	Ave.	Min.	Max.
Oct. '55				0.21	0.02	1.3				0.18	0.08	0.23
Jan. '56	0.02	0.02	0.03	0.07	0.01	0.12				0.02	0.01	0.04
Feb.				0.03	0.02	0.05				0.03	0.01	0.09
March	0.02	0.00	0.05	0.02	0.01	0.03				0.01	0.00	0.01
April	0.01	0.00	0.02	0.01	0.00	0.02				0.05	0.01	0.24
May	0.01	0.00	0.02	0.01	0.00	0.02				0.04	0.01	0.17
June	0.01	0.00	0.02	0.00	0.00	0.00				0.01	0.00	0.17
July	0.00	0.00	0.00	0.00	0.00	0.00				0.10	0.00	0.41
Aug.	0.01	0.00	0.01									
Sept.	0.01	0.01	0.01									
Oct.	0.02	0.02	0.03	0.01	0.01	0.01				0.08	0.06	0.23
Dec.										0.19	0.06	0.20
Jan. '57				0.07	0.01	0.11	2.6	2.2	3.0	0.07	0.00	0.30
May							0.05	0.05	0.05	0.05	0.02	0.10
Dec.				0.00	0.00	0.00						

It was at this stage that industrial hygiene studies were instituted to evaluate worker exposures to TDI vapors. During the next 17 months, more than one thousand air samples were collected and analyzed for TDI in cooperation with the plant chemist.

The initial steps in this study involved a classification of jobs in the polyurethane processes. In general, these jobs and work stations became the air sampling stations for evaluating exposures.

During the period of this study there were many changes in plant ventilation, operating procedures and process equipment. These changes introduced factors which affected exposure results. In recent months, the general trend of average TDI concentrations in air has been downward.

The chemical methods used for TDI determination were those suggested by representatives of the du Pont Company. The early studies involved air sampling in a Cellosolve (ethylene glycol monoethylether) and sodium nitrite solution which produced a yellow to amber color upon reaction with TDI. Color comparisons were made with the spectrophotometer at 425 millimicrons.[4]

More recently, air samples were collected in a dilute acid solution consisting of 22.7 ml of glacial acetic acid and 33.3 ml of hydrochloric acid per liter of solution. Standards were prepared from a solution containing 2.2 mg of TDI per liter of 0.6*N* acetic acid. Color was developed by diazotization with an organic reagent. The concentration of the colored complex was determined by measuring optical density at 550 millimicrons.[5]

All air samples were collected using a modified midget impinger apparatus. The sampling rate was reduced to approximately one liter per minute by a limiting orifice in the line between the impinger flask and pump.

Hygienic Concentrations for TDI

In 1955, Swensson[2] et al. reported that the chronic toxicity of TDI was of a low order. However, on the basis of industrial operations, they also reported that vapors of the chemical had unusual irritating properties involving the eyes, throat and respiratory tract.

Zapp[1] has suggested a hygienic limit of 0.1 ppm for continuous exposure to TDI vapor. The results of our studies tend to verify the need for a very low exposure limit, but we found no direct correlation between degree of exposure and the appearance of respiratory symptoms.

Study Results

Tables IA and IB show the average and range of concentrations of TDI vapors at various operations over the period of this study. These results are further summarized in graphic form in Figure 5, which illustrates the average monthly concentration of TDI vapor throughout the entire plant. The highest vapor concentration found was 2.2 ppm.

During the study period, the plant physician reported a total of 83 illnesses related to the polyurethane operations which required medical attention. These cases were classified as follows:

Upper respiratory infection	54
Tracheitis	11
Bronchitis	9
Bronchial asthma	9
TOTAL	83

Of the 83 cases, seven were hospitalized for periods ranging from one to 49 days. Total hospitalization amounted to 79 days. In addition, a large number of minor cases were treated in the plant dispensary.

An analysis of the medical records revealed that most cases of illness appeared in workers between the third and fourth week after assignment to the polyurethane departments. It also revealed a lack of correlation between the magnitude of TDI vapor exposure and the appearance of respiratory cases. The probable reasons for this lack of correlation are believed to be:

(1) Individual susceptibility to respiratory irritation.

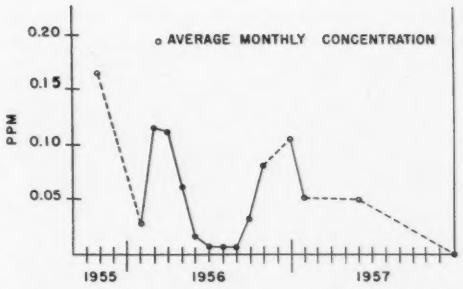

FIGURE 5. Graph showing average monthly concentration of TDI vapor in entire plant. Broken line indicates periods during which monthly determinations were not made.

(2) Short exposures to relatively high TDI concentrations during periods of generally low average concentrations. It is recognized that the integrating sampling method used would not indicate short peak concentrations.

(3) Inadequate records of absenteeism and mild respiratory irritation.

There was indication that once workers were affected by the TDI exposure, they could not tolerate even minute re-exposures to TDI. However, the plant involved in these cases made it a policy not to relocate workers in these departments after a reported illness.

During the 15 months following the major study period, 16 cases of illness were reported from the polyurethane departments. With the exception of an occasional high exposure through

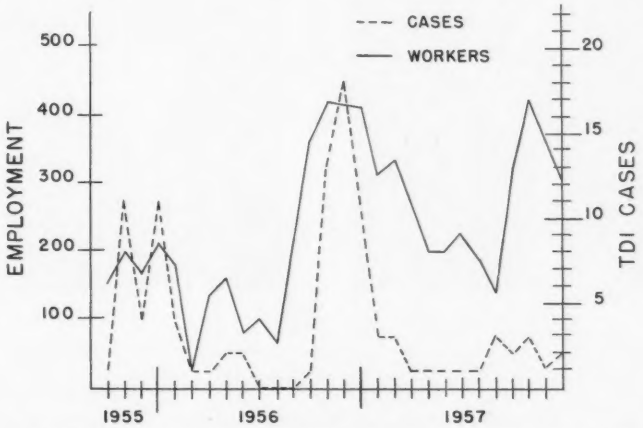

FIGURE 6. Graph comparing number of workers exposed to TDI with total number of cases treated monthly by the physician.

careless operation or malfunctioning equipment, average TDI concentrations had been reduced considerably. This reduction in concentration was reflected by the decrease in total number of TDI cases in relation to the number of workers employed (Figure 6).

Summary

1. Specific industrial operations using polyurethane chemicals have been described.

2. The results of TDI vapor exposures in these operations have been reported.

3. A brief medical report involving cases of respiratory irritation among workers in these operations has been given.

4. It was observed that most of the illnesses occurring among workers in the polyurethane departments appeared after three to four weeks of exposure to TDI.

5. Probable reasons for the lack of correlation between the magnitude of TDI vapor exposure and the appearance of respiratory cases are discussed.

6. A downward trend in the magnitude of TDI exposures and in the number of TDI cases has been shown.

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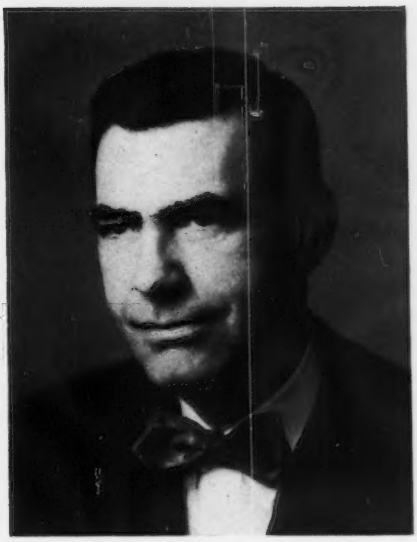
MICHIGAN SECTION AWARD

T. R. Torkelson

F. Oyen

D. D. McCollister



V. K. Rowe

THE MICHIGAN SECTION, AIHA, made the second annual Michigan Section Award for the outstanding paper appearing in the AIHA Journal during the preceding year at the annual dinner meeting in Chicago, April 29, 1959. The recipients were T. R. Torkelson, F. Oyen, D. D. McCollister and V. K. Rowe for their paper, "Toxicity of 1,1,1-Trichloroethane as Determined on Laboratory Animals and Human Subjects" (p. 353, Oct., 1958). The award of cash and certificates of recognition, presented by V. J. Castrop for the Michigan Section, was accepted by T. R. Torkelson on behalf of the authors.

Lightweight Air-Sampling Pumps for Field Use

MORTON LIPPMANN, EMIL E. CHRISTOFANO, and ROBERT T. GRAVESON

Health and Safety Laboratory, U.S. Atomic Energy Commission, New York Operations Office

Introduction

THE HEALTH and Safety Laboratory (HASL) routinely conducts comprehensive air contamination surveys during which up to a thousand air samples may be collected in a weeks time. Such surveys are often at locations hundreds or thousands of miles from the Laboratory. It has been our experience that unless the air-sampling pumps used are reliable, rugged, light in weight, easy to service, convenient to use, and of sufficient capacity to draw adequate samples for analysis in a reasonable length of time, the progress of the survey will be retarded. Since no commercially available pumps satisfied enough of these criteria to satisfy the HASL requirements, two new lightweight pumps have been designed. One is a 110-volt pump which will draw up to 30 liters per minute and the second is a battery-powered pump which will draw up to 14 liters/min.

Description of 110-Volt Pump

Figure 1 shows the complete 110-volt pump. A removable 1⅛-inch filter head is attached through a quick-connect lock fitting made from Becton-Dickinson Leur-Lok hose adaptors.* The filter head can be replaced quickly with another head or with a female hose adaptor connected to a remote sampling device through rubber tubing. The pump is 4 x 7 x 4½ inches overall, and

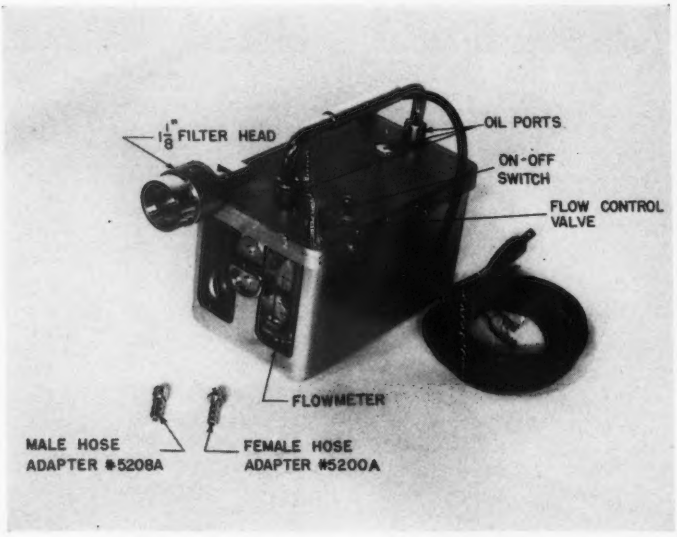


FIGURE 1. Complete 110-volt pump with 1⅛-inch filter head attached through quick-connect fitting.

* Hose adaptors 5200A and 5208A available from Becton-Dickinson, Rutherford, New Jersey. The male fitting on the pump was made from a 5208A hose adaptor. The hose end was cut down, threaded and screwed into a corresponding threaded hole in the inlet fitting. Similarly, hose adaptor 5200A was modified and attached to 1⅛" filter head.

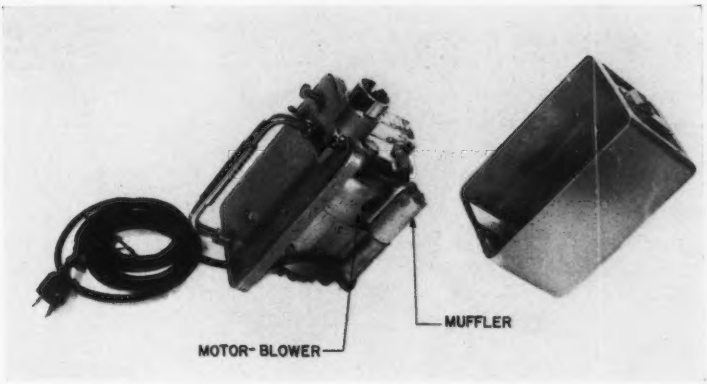


FIGURE 2. 110-Volt pump removed from casing to show the positions of the components.

weighs 6 pounds 2 ounces. Figure 2 shows the pump removed from the casing. The motor-blower is a modified sliding-vane compressor-motor produced by the Dumore Company for use in paint sprayers. The muffler on the discharge was designed by HASL[1], and reduces the noise level from 39 to 23 db above room sound level. The flow is metered through a 15–35 liter/min rotameter with an integral flow control valve. Copper tubing is used for connecting air lines within the pump. The pump is suitable for continuous duty if lubricated after each eight hours of operation. Oil is introduced through three oil taps located conveniently in the top of the case (see Figure 1).

The pump characteristics are indicated in Figure 3. The pump will easily draw one cfm (28.3 liter/min) through a Greenburg-Smith impinger or a one-inch circle of Whatman #41 filter paper.

Description of Battery-Powered Pump

Figure 4 shows the complete battery-powered pump. The pump, rotameter, 1⅛-inch filter head, charging cord, and rubber tubing all fit into a leather carrying case, which is 7½ x 4¼ x 5½ inches. The complete unit including batteries and charger weighs 9 pounds 2 ounces. Figure 5 shows the pump removed from the carrying case. The battery compartment cover is removed to help illustrate the location of the various components, including silicon rectifiers, step-down transformer, batteries, and motor-blower. The quick-connect lock fittings for external air connections are the same as those used for the 110-volt pump.

The motor-blower is the Gast Pump Model AD 440, which was originally designed for the HASL.[1] The batteries are three 20-amp-hr silver-zinc cells. The battery compartment will also accommodate four 5-amp-hr nickel-cadmium cells. While for similar weight and space these nickel-cadmium batteries will run the pump for only 15% as long as the silver-zinc cells, they can be charged through many more cycles, will last almost indefinitely, and cost about half as

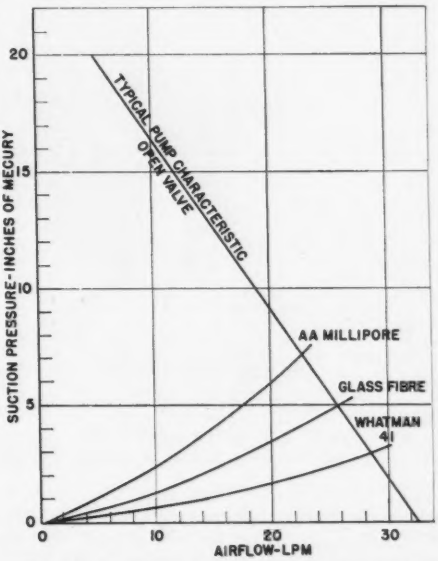


FIGURE 3. Airflow rate versus suction pressure for 110-volt pump and for 1-inch circles of filter paper in 1⅛-inch HASL filter heads.

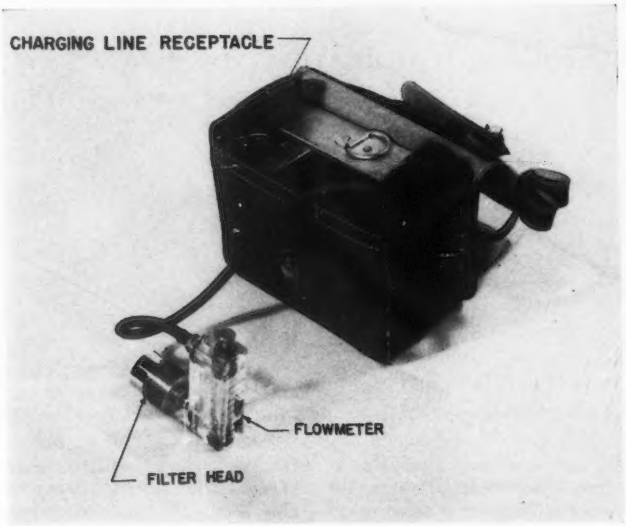


FIGURE 4. Complete battery-powered pump.

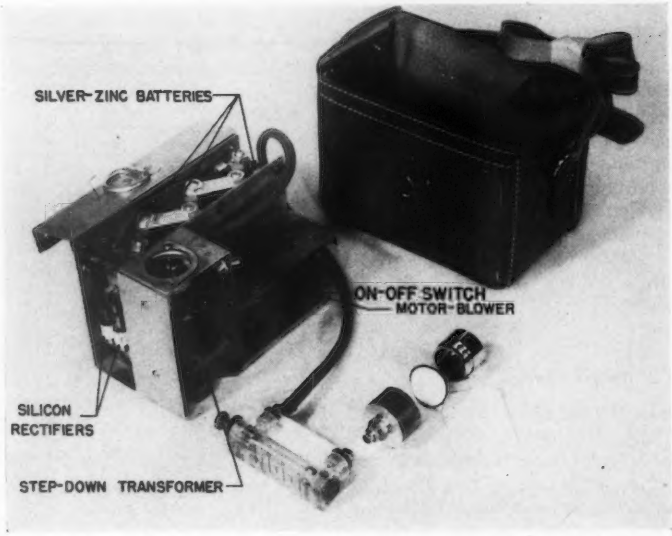


FIGURE 5. Battery-powered pump is removed from carrying case and the battery compartment cover is displaced to show the positions of the components.

much. Silver-zinc cells deteriorate after about one year. Thus, if the sampler is used only occasionally, nickel-cadium cells may be preferable.

The pump characteristics are indicated in Figure 6. It can be seen that the pump has more than enough capacity to operate the usual bubbler and wet scrubber equipment, and midget impingers. It also will draw a generous sample through a 1- or 2-inch filter. Figure 7 is a plot of flow rate vs time for the pump with an overnight charge of 16 hours, and with a full charge of 48 hours. After a full charge, the pump can be used for seven continuous hours of sampling. The silver-zinc cells may be recharged

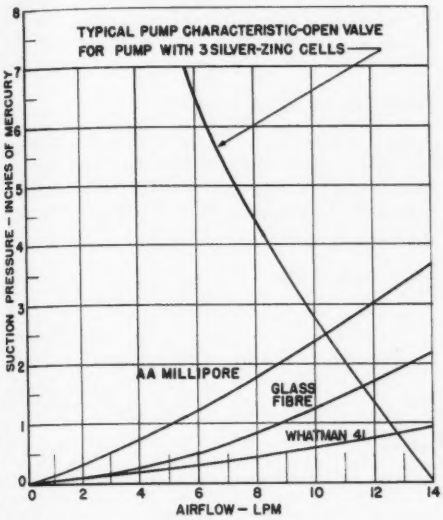


FIGURE 6. Airflow rate versus suction pressure for battery-powered pump with three 20-amp-hr silver-zinc cells, and for 1-inch circles of filter paper in 1⅛-inch HASL filter heads.

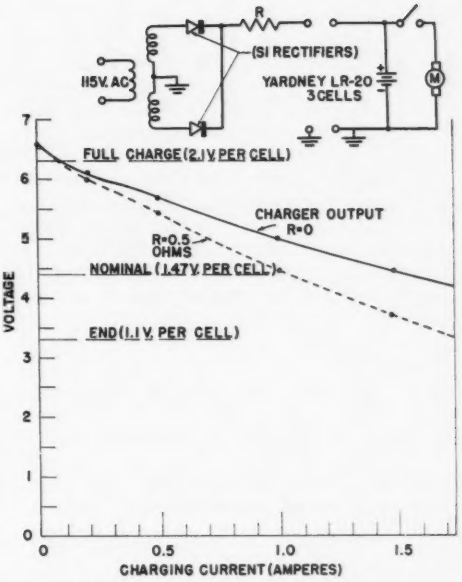


FIGURE 8. Charger circuit diagram and voltage-current characteristics of silver-zinc cells.

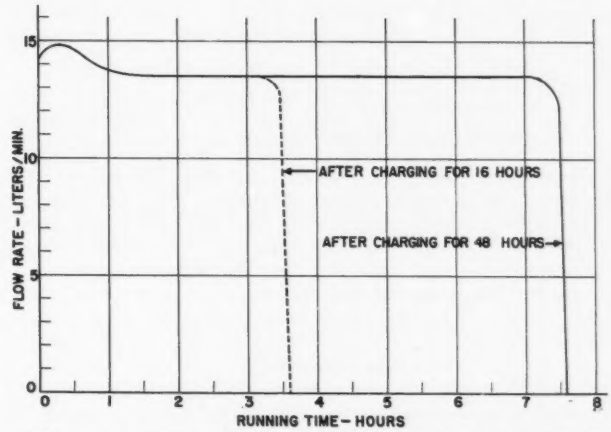


FIGURE 7. Airflow rate versus operating time for battery-powered pump with three 20-amp-hr silver-zinc cells. The solid line represents the discharge following a full 48-hour charge. The dotted line represents the discharge following an overnight 16-hour charge.

from 60 to 100 times within a one-year useful life. Using nickel-cadmium cells, the delivery is substantially the same. After an overnight charge, it is possible to sample for something over an hour. These cells may be recharged almost indefinitely.

Both battery types use only enough electrolyte to dampen the plates; consequently, spillage cannot occur. The electrolyte level seldom requires adjustment, and the cells may be stored in a discharged condition without harm.

Each silver-zinc cell will deliver 2.1 volts when fully charged. This rapidly falls to 1.5 volts where it is maintained over much of the discharge cycle. When the delivered voltage falls to 1.1 volts, very little useful life remains, and

the voltage then drops to zero in a few minutes. Three silver-zinc cells will deliver 6.3 volts at full charge, falling within half an hour to 4.4 volts, which is maintained for about seven hours. The voltage then drops to 3.3 volts within about 10 minutes, at which time regeneration is necessary.

The built-in charging circuit has a full-wave rectifier and root mean square transformer. The circuit diagram for the charger and the voltage vs current characteristic for the silver-zinc batteries are shown in Figure 8. A 0.5 ohm resistor is used to control the charging rate at one ampere when the battery is at 1.5 volts per cell. The initial charging rate of 1.75 amperes tapers to one ampere for the plateau region. As the battery approaches full charge the rate drops to a trickle of 0.2 ampere. With this circuit it is not possible to overcharge the silver-zinc cells.

Four nickel-cadmium cells will deliver 5.1 volts at full charge, 4.9 volts over the effective discharge cycle, and 4.6 volts at discharge. With the charger described above, the battery is recharged at 0.75 ampere which tapers to 0.6 ampere at full charge. Here too, the battery is not damaged by charging for too long a period.

Construction Details

Both pumps were designed and constructed in the Health and Safety Laboratory, and complete engineering drawings are available upon request. Commercial versions of both pumps are now available.

Acknowledgments

Grateful acknowledgement is made to W. B. Harris and H. D. LeVine for their contributions to the design of these pumps, and to Alfred Bruzzone, who constructed the prototype models and made many useful design improvements.

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Opportunity Knocks

Another milestone in the expanding publications of AIHA will soon be passed with the offering for sale of a limited number of complete sets of bound volumes of the American Industrial Hygiene Association Quarterly from 1940 through 1957 (Volumes 1 through 18). A number of back issues have been collected and, where necessary, issues are being reprinted to provide full sets of uniform size for binding. These sets will be in hard binding covered with black Fabrikoid and lettered in gold.

A cumulative index for the entire series (Volumes 1 through 18) is in preparation and will be printed and bound with each set. This will be a particularly valuable feature of these sets.

The price is $250.00 per set. A small number of sets are still available; orders should be sent to American Industrial Hygiene Association, 14125 Prevost, Detroit 27, Michigan.

Vapor Toxicity of Allyl Chloride as Determined on Laboratory Animals*

T. R. TORKELSON, M.S., M. A. WOLF, M.S., F. OYEN, and V. K. ROWE, M.S.

Biochemical Research Laboratory, The Dow Chemical Company, Midland, Michigan

Introduction

THE USEFULNESS of allyl compounds as intermediates in chemical synthesis is becoming more widely known and their use is increasing yearly. This increase in usage makes additional physiological data desirable so that their hazard to health can be evaluated better. Toxicological and pharmacological data are available in the literature concerning the acute toxicity of 3-chloro-1-propene, more commonly known as allyl chloride. The acute vapor toxicity was reported to be high by Adams, Spencer and Irish[1] and moderate by Smyth and Carpenter.[2] These later workers also reported that liquid allyl chloride was slightly irritating to the eyes and skin; that it was moderate in acute oral toxicity, a range finding acute oral LD_{50} of 0.7 g/kg having been determined for rats; and that the material was absorbed through the skin although large amounts were required to cause death, an LD_{50} of 2.2 g/kg being determined for 24-hour skin absorption on rabbits. A summary of hazards based on industrial experience has also been published.[3] However, data describing the results of repeated inhalation studies apparently are not available. Smyth[4] reported that the ACGIH Threshold Limit Value of five ppm was based on the result of single exposures to laboratory animals and by analogy to chloroprene.

The work reported herein was undertaken to provide a basis for judging the hazards associated with repeated exposures and will describe the results of experiments designed to determine the effect of daily inhalation of low levels of allyl chloride on laboratory animals. It will also describe the results of human sensory perception at three ppm. The following article in this same journal will report the results of similar experiments using allyl alcohol.

* Presented at the Twentieth Annual Meeting of the American Industrial Hygiene Association, Chicago, Illinois, April 30, 1959.

Experimental

PHYSICAL PROPERTIES

Allyl chloride is a clear liquid with a very pungent smell. It has a boiling point of 45°C. at 760 mm Hg and a specific gravity of 0.9376 at 20/4°C. Allyl chloride is flammable (TOC Flashpoint is −20°F) and the explosive limits are reported to be 3.28 to 11% by volume. The sample used in this study was of high purity. Analysis by vapor phase chromatographic technique reported no impurities boiling below 100°C.

EXPERIMENTAL TECHNIQUE

Eight ppm (0.025 mg/liter): Because of the lack of data relative to the chronic toxicity of allyl chloride, a pilot experiment was conducted in which small groups of animals were exposed repeatedly to an average concentration of eight ppm in air. A total of 28 seven-hour exposures were made during a 35-day period, the exposures being made on the basis of a five day week. Two matched groups of animals were selected from the stock colony of this laboratory. Each group consisted of five rats of each sex, four male guinea pigs, and one female rabbit. One group was exposed to the chemical in a glass-walled chamber (160-liter capacity) using metering equipment of a type previously described.[5] The second group was placed in a similar chamber and exposed daily to room air under similar conditions. The animals were transferred daily to expanded metal cages with hinged covers and placed in the exposure chamber during the exposure period. The animals were fed commercial diet[6] and water *ad lib*, except during their exposure periods. Guinea pigs were given cabbage in addition to the commercial diet. The animals were weighed twice a week at the beginning of the experiment and once a week after it was evident that the weights were not being affected by the two day withdrawal from exposure on week ends. Animals were observed frequently for general appearance and be-

havior. Growth and mortality records were kept for each group. Failing animals were killed for examination. At autopsy, the rats were starved overnight and all animals were killed by decapitation on the day following their last exposure. All animals were observed for gross pathological changes. The lungs, hearts, livers, kidneys, spleens and testes were removed and weighed. Portions of these organs, as well as the pancreas and adrenals, were preserved in formalin. Sections were made and stained with hematoxylin-eosin for histological examination. Where applicable, the "t" test[7] was used in comparing mean values of body weights and of organ weights, probability values (P) of 0.05 or less being interpreted as a significant difference.

Analysis of the chamber air was made by drawing samples directly from the chamber through saran plastic tubing and burning the sample by passing it through a quartz tube filled with platinum gauze and heated by an electric furnace to 1000°C. The resulting chloride was trapped in 25 ml of a solution containing one per cent sodium formate and one per cent sodium carbonate. The scrubber solution was transferred with repeated rinsings to a 50 ml volumetric flask, made up to volume, and subsequently titrated using a micro-Volhard technique.[8] Analytical values ranged from 7.9 to 10 ppm with the majority of the values falling between 8.0 to 8.5 ppm. In addition, the chamber concentration was continuously monitored by a continuous combustion-conductivity analyzer.[9] An analysis of the charts obtained showed no deviations of significance during the course of the experiment.

Three ppm (0.009 mg/liter): On the basis of the pilot experiment, a larger scale experiment was set up using three groups of animals which were selected on the basis of age and weight. Each group consisted of twenty four rats, three rabbits, nine guinea pigs and one dog of each sex. The first group was exposed to three ppm of allyl chloride in air, seven hours per day, five days per week, for a total of 127 to 134 exposures in 180–194 days. The second group received seven-hour exposures to room air in a similar chamber. The third group was held in the animal quarters as unexposed controls.

The source and selection of the rats, rabbits and guinea pigs and their feeding were basically the same as in the previous experiment. The dogs were purebred beagles obtained from a local kennel and kept in the animal quarters for several weeks to assure their good health and acclimatization. They were given 1/3 to 1/2 can of commercial dog food[10] each day in addition to their dry laboratory diet.[6] During the experiment, the rats, guinea pigs, and rabbits receiving exposures, were housed in stainless steel cages, mounted on racks which were rolled into the exposure chamber each day. The rats were housed four to a cage, the guinea pigs two to a cage, and the rabbits and dogs singly. One rack held the guinea pigs and rabbits, the other the rats and dogs. Each morning the dogs were transferred from large holding cages to smaller cages mounted on the racks. All food and water were removed during the exposure to prevent absorption of the chemical and subsequent ingestion by the animals. Food and water were not removed from the unexposed controls.

The chambers used in this study were of a different design than those which we have previously described. They were of a vault type, constructed of stainless steel with two hinged doors opening from the center on the front. Their volume was about 3,700 liters. The two roll-in racks were pushed up on tracks into the chamber each day. Doors were sealed with Silastic® gasketing. Each chamber was equipped with its own air pump, a circulating fan and temperature controlled metering equipment to deliver the toxicant into the air stream. An air-flow of 340 liters/min was passed through the chambers. The air entered the top of the chamber through a two-inch pipe and was exhausted through a manifold extending across the back and along each side of the chamber, about six inches from the floor. A large air circulating fan located immediately in front of the intake was used to assure even distribution and to prevent channeling of the air through the chamber.

Analysis of the chamber atmosphere was made by the method described above. Values averaged 2.9 ppm with a range of 1.8 to 3.9. The bulk of the values, however, fell between 2.6 and 3.5 ppm. The chamber concentration was monitored continuously by the combustion-conductivity analyzer and no deviations of significance in concentration were noted.

The procedure at autopsy was basically the same as that described for the study at eight ppm with the following additions: (1) The dogs were killed by exsanquination after being anesthetized with Thiopental Sodium (Pentothal, Abbott). (2) Samples of blood from representative groups of animals were collected for determination of terminal blood-urea-nitrogen (BUN), and total blood-non-protein-nitrogen (NPN) as well as for hematological examination. (3) The male and female rats were split into two matched groups on the basis of original body weight; one group was killed along with the rest of the animals the day following their last exposure; and the other group was held for two months as a recovery group and then killed. Since the only

change seen in animals killed immediately after exposure was in the liver of the female rats, examination of the recovery group was directed toward the liver and kidneys. Only these organs were weighed and saved for microscopic examination.

Human Sensory Tests: Thirteen human volunteers evaluated the presence or absence of characteristic odor of allyl chloride at the level of three ppm. This was done by having groups of two or three volunteers quickly enter the large chamber previously described, after analysis

TABLE I

Summary of Final Average Body and Organ Weights of Animals Receiving Repeated 7-Hour Exposures to 8 ppm Allyl Chloride Five Days Per Week for Five Weeks

Species	Group	Sex	No. of surviving animals	Body weight, gm	Organ weights, gm/100 gm body weight					
					Lung	Heart	Liver	Kidney	Spleen	Testes
Rat	8 ppm allyl chloride	M	5	287	0.66	0.38	2.95	0.77	0.43	1.06
	air-exposed control	M	5	256	0.60	0.39	2.81	0.77	0.44	1.17
	8 ppm allyl chloride	F	5	183	0.70	0.36	2.68	0.77	0.37*	—
	air-exposed control	F	5	179	0.67	0.38	2.74	0.72	0.46	—
Guinea pig	8 ppm allyl chloride	M	4	508	0.68	0.36	3.97	0.84	0.11	0.48
	air-exposed control	M	4	496	0.67	0.34	4.20	0.80	0.13	0.49
Rabbit	8 ppm allyl chloride	F	1	2520	0.41	0.22	2.73	0.49	0.04	—
	air-exposed control	F	1	3120	0.40	0.20	3.09	0.47	0.04	—

* This variation is statistically significant (See text).

TABLE II

Mortality and Its Cause Among Animals Receiving Repeated 7-Hour Exposures to 3 ppm Allyl Chloride Five Days Per Week for Six Months

Species	Sex	Group	No. of animals started	No. of deaths	Reason for sacrifice or cause of death
Rat	M	3 ppm allyl chloride	24	4	1—Ear infection 1—Lung infection 2—Found dead, cause not known
	M	air-exposed control	24	4	3—Lung infection 1—Not known
	M	unexposed control	24	3	2—Lung infection 1—Not known
	F	3 ppm allyl chloride	24	1	1—Pericarditis
	F	air-exposed control	24	0	—
	F	unexposed control	24	1	1—Lung infection
Guinea pig	M	3 ppm allyl chloride	9	0	—
	M	air-exposed control	9	0	1—Lost
	M	unexposed control	9	1	1—Found dead, cause not known
	F	3 ppm	9	0	
	F	air-exposed control	9	1	1—Found dead, cause not known
	F	unexposed control	9	2	2—Pneumonia
Rabbit	M	3 ppm allyl chloride	3	1	1—Not known
	M	air-exposed control	3	2	1—Not known 1—Lung infection
	M	unexposed control	3	0	—
	F	3 ppm allyl chloride	3	0	—
	F	air-exposed control	3	0	—
	F	unexposed	3	0	—
Dog	M	3 ppm allyl chloride	1	0	—
	M	air-exposed control	1	0	—
	M	unexposed control	1	0	—
	F	3 ppm allyl chloride	1	0	—
	F	air-exposed control	1	0	—
	F	unexposed control	1	0	—

showed that the concentration was that which was desired. They remained in the concentration for one to three minutes. Independently, each gave a verbal report to the tabulator.

Results

Eight ppm (0.025 mg/liter): The male and female rats, male guinea pigs and female rabbit, which were subjected to 28 seven-hour exposures to eight ppm of allyl chloride, showed no evidence of ill effect when judged by growth, behavior, mortality, gross appearance and average final body weights. The female rats were the only animals which showed any variation in organ weights, and this only in average spleen weights (Table I). This finding appears to be of little importance however, as there were no supporting histopathological findings in the spleen. Histopathological examination however, did show significant evidence of ill effect of a severe degree in the liver and kidneys of essentially all the animals. These were characterized by dilation of the sinusoids, cloudy swelling and focal necrosis in the liver; and by changes in the glomeruli, necrosis of the epithelium of the convoluted tubules and proliferation of the interstitial tissues in the kidneys.

Three ppm (0.009 mg/liter): Summaries of the mortality, growth and organ weight data are shown in Tables II and III, and values found for blood-urea-nitrogen and blood-non-protein-nitrogen are shown in Table IV. Hematological values are summarized in Table V. The animals of both sexes that received 127 to 134 seven-hour exposures in a period of 194 days to three ppm allyl chloride showed no evidence of ill effect when judged on the basis of growth, behavior, mortality, gross appearance and final body weight. The terminal values reported for BUN and NPN (Table IV) were in several cases below those found for the control animals for this experiment. They were however, within the normal limits for control animals from the same stock used in previous experiments and in the case of dogs are also within the normal limits listed by Dukes[11] (BUN 10–20 mg/100 ml; NPN 17–38 mg/100 ml). In the absence of pathology in the liver and kidneys which might be expected if differences in NPN and BUN were due to exposure to allyl chloride and since the values lie

TABLE III

Summary of Final Average Body and Organ Weights of Animals Receiving Repeated 7-Hour Exposures to 3 ppm Allyl Chloride Five Days Per Week for Six Months

Species	Sex	Group	No. of surviving animals	Body weight, gm	Organ weights, gm/100 gm body weight					
					Lung	Heart	Liver	Kidney	Spleen	Testes
Rat	M	3 ppm allyl chloride	9	386	0.57	0.33	2.57	0.69	0.26	0.86
	M	air-exposed control	10	375	0.52	0.34	2.50	0.73	0.27	0.85
	M	unexposed control	12	371	0.53	0.32	2.59	0.70	0.24	0.83
	F	3 ppm allyl chloride	11	228	0.68	0.37	2.71	0.75	0.34	—
	F	air-exposed control	12	220	0.64	0.34	2.77	0.75	0.32	—
	F	unexposed control	12	215	0.64	0.39	2.70	0.76	0.28	—
Guinea pig	M	3 ppm allyl chloride	9	908	0.54	0.26	2.96	0.69	0.10	0.52
	M	air-exposed control	9	946	0.49	0.27	3.16	0.66	0.10	0.52
	M	unexposed control	8	933	0.55	0.26	3.31	0.70	0.11	0.53
	F	3 ppm allyl chloride	9	880	0.55	0.27	3.71	0.71	0.11	—
	F	air-exposed control	8	875	0.52	0.26	3.72	0.75	0.13	—
	F	unexposed control	7	860	0.67	0.29	3.64	0.77	0.12	—
Rabbit	M	3 ppm allyl chloride	2	2920	0.41	0.18	2.20	0.48	0.02	0.19
	M	air-exposed control	1	3160	0.31	0.20	2.53	0.52	0.02	0.22
	M	unexposed control	3	3200	0.35	0.29	1.77	0.47	0.02	0.22
	F	3 ppm allyl chloride	3	3670	0.31	0.19	2.03	0.44	0.03	—
	F	air-exposed control	3	3570	0.43	0.23	2.05	0.46	0.04	—
	F	unexposed control	3	3650	0.32	0.18	1.75	0.38	0.03	—
Dog	M	3 ppm allyl chloride	1	9500	1.03	0.85	2.54	0.44	0.48	0.22
	M	air-exposed control	1	11300	0.86	0.98	2.65	0.44	0.25	0.18
	M	unexposed control	1	14100	0.77	0.67	2.66	0.42	0.20	0.17
	F	3 ppm allyl chloride	1	11200	1.12	0.78	1.98	0.39	0.33	—
	F	air-exposed control	1	12200	1.08	0.91	2.43	0.37	0.36	—
	F	unexposed control	1	11000	0.76	0.81	2.31	0.45	0.21	—

within normal limits, the apparent changes were not considered to be significant or a result of the exposure to the chemical. The only deviation of significance in organ weights or hematological values were for the male dog. The elevated eosinophil count (10%) and the increased spleen weight (0.48 g/100 g body weight) reported for this animal were probably the result of the slight interstitial pneumonitis and a peri-arteriolar granuloma which were seen when a section of the lung was examined histologically. The eosinophil level and the spleen weight were not considered to be due to the compound but to a concurrent infection. No significant changes were seen on gross pathological examination. The only finding of importance seen during histopathological examination was a slight change in the livers of the female rats killed immediately after exposure. This change, consisting of slight central lobular degeneration, was of a type often seen in control groups. But since it was not seen in the male rats nor in any other species and was not present in any of the rats allowed to recover for two months, it was considered an effect of the exposure.

Human Sensory Tests: Ten of the thirteen human volunteers reported a definite odor but no irritation when exposed to three ppm of allyl chloride for one to three minutes.

TABLE IV

Average Values for Blood-Urea-Nitrogen and Blood-Non-Protein-Nitrogen For Animals Receiving Repeated 7-Hour Exposures to 3 ppm Allyl Chloride Five Days Per Week for Six Months

Species	Group	Sex	No. of animals	BUN mg/100 ml	NPN mg/100 ml
Rat	3 ppm allyl chloride	F	7	20.8	46.0
	air-exposed control	F	8	21.4	46.5
	unexposed control	F	8	20.2	49
Guinea pig	3 ppm allyl chloride	M	5	26.9	—
	air-exposed control	M	5	26.7	—
	unexposed control	M	4	37.7	—
	3 ppm allyl chloride	F	4	17.7	—
	air-exposed control	F	5	28.3	—
	unexposed control	F	4	30.4	—
Rabbit	3 ppm allyl chloride	M	2	20.2	—
	air-exposed control	M	1	16.9	—
	unexposed control	M	1	18.8	—
	3 ppm allyl chloride	F	2	24.1	—
	air-exposed control	F	3	21.9	—
	unexposed control	F	3	24.8	—
Dog	3 ppm allyl chloride	M	1	12.6	34
	air-exposed control	M	1	21.2	42.5
	unexposed control	M	1	22.5	46
	3 ppm allyl chloride	F	1	11.6	35.5
	air-exposed control	F	1	22.0	38
	unexposed control	F	1	21.2	Contaminated

TABLE V

Average Hematological Values for Dogs Receiving Repeated 7-Hour Exposures to 3 ppm Allyl Chloride Five Days Per Week for Six Months

Group	When sampled	Sex	No. of animals	WBC × 10^3	Hb gm/100 ml	Hematocrit	Differential count per cent				
							Neutrophils	Lymphocytes	Monocytes	Eosinophils	Basophils
3 ppm allyl chloride	preexposure	M	1	15.0	13.0	44	53	45	1	1	0
	terminal	M	1	12.2	15.0	53	47	41	2	10	0
Air-exposed control	preexposure	M	1	10.1	12.0	42	42	52	4	2	0
	terminal	M	1	10.3	14.0	51.5	48	49	1	2	0
Unexposed control	preexposure	M	1	21.4	13.0	44.5	57	42	0	1	0
	terminal	M	1	15.8	13.5	50	49	45	1	5	0
3 ppm allyl chloride	preexposure	F	1	12.3	15.0	50	47	49	3	1	0
	terminal	F	1	11.0	13.5	49	36	58	3	3	0
Air-exposed control	preexposure	F	1	19.4	11.0	38	52	47	1	0	0
	terminal	F	1	14.8	14.5	49	56	38	3	3	0
Unexposed control	preexposure	F	1	22.9	14.0	45	48	46	3	3	0
	terminal	F	1	16.7	13.0	47	52	43	3	2	0

Significance of Results

Vapor Inhalation—Repeated Exposures: Allyl chloride has been shown to be one of the more toxic chlorinated hydrocarbons when inhaled repeatedly. Results of the experiments reported herein show that repeated seven-hour daily exposures to eight ppm caused liver and kidney injury in all three species studied. Exposure of animals to three ppm was found to cause what may be interpreted as a border line effect, slight reversible liver pathology in female rats only. Hence, a level of three ppm offers no margin of safety and exposures must necessarily be limited to less than three ppm if the probability of injury is to be minimized.

Industrial Hygiene Standard: On the basis of the above data, an industrial hygiene standard of two ppm is suggested for allyl chloride. This standard as used here, is defined as that concentration of allyl chloride below which practically all fluctuations must fall during a working day where repeated, prolonged human exposure is likely. Because two ppm appears to offer a very small margin of safety, it is recommended that the time-weighted average of daily exposures (all below two ppm) not exceed 1.0 ppm. Under such conditions, the probability of injury would seem to be remote.

Human Sensory Response: The human sensory tests indicate that most people would be able to detect the presence of three ppm of allyl chloride. However, the warning properties are very inadequate insofar as providing any degree of protection where repeated exposure is likely. Chemical analysis of the air, rather than human senses, should be used in controlling the concentration in the air.

Precautions for Safe Handling

The following precautions for safe handling can be made on the basis of industrial experience, previous data and the data presented herein.

Inhalation: The concentration of allyl chloride in the air of work areas should be maintained below two ppm with a time-weighted average of all such values equalling no more than 1.0 ppm. Concentrations of allyl chloride in this range do not have warning properties, but may be detectable by odor by some people. Suitable analytical methods rather than sensory perception should be depended upon for control.

Concentrations which are likely to be injurious in short periods of time will be painful to the eyes and nose. The warning odor should not be ignored.

To prevent ill effects from inhalation, allyl chloride should be handled in closed systems or with respiratory protection unless adequate ventilation is provided. Natural ventilation should not be depended upon even in outdoor areas. If spills should occur, the area should be evacuated at once and re-entered only after thorough aeration. If it is necessary to re-enter before aeration is completed, adequate respiratory protection such as a full face gas mask equipped with an organic vapor (black) canister, an air or oxygen supplied respirator, or a self-contained breathing apparatus should be worn.

Eye Contact: The eyes must be protected if the likelihood of contact exists. Tight fitting chemical workers' goggles or preferably a full-face gas mask should be worn to prevent getting the material in the eyes.

Skin Contact: Protective clothing impermeable to the material should be worn where the likelihood of skin contact exists. Allyl chloride should never be allowed to remain on the skin. Clothing and shoes should not be allowed to become contaminated with the material, if they do they should be promptly removed and not worn again until completely free of the material.

Ingestion: All containers should be properly labelled so that ingestion due to mistaken identity cannot occur. Hands should be washed with soap and water before eating or smoking.

Summary

Laboratory animals were exposed repeatedly to either eight or three ppm of allyl chloride in air to establish conditions safe for repeated exposure. Rats, guinea pigs and rabbits had definite liver and kidney pathology after one month of repeated seven-hour exposures to eight ppm. These species and dogs tolerated similar exposures to three ppm for six months with only slight reversible liver pathology seen in female rats.

A standard for evaluating regular seven- to eight-hour daily exposures may be defined as the concentration below which practically all analytical results must fall. The value of two ppm is suggested as this standard for allyl chloride, with the proviso that the time-weighted average for all exposures not exceed 1.0 ppm.

Handling precautions for industrial usage are described.

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Gordon Research Conferences

THE GORDON RESEARCH CONFERENCE on Toxicology and Safety Evaluations will be held August 10 through 14, 1959, at Kimball Union Academy, Meriden, New Hampshire. Don D. Irish is Chairman and David W. Fassett is Vice Chairman. Discussion periods will be devoted to the following topics or areas: Physical-Chemical Factors in the Development of Barrier Materials; Pulmonary Tissue Changes Induced by Inhaled Industrial Dust; Deriving Tentative Threshold Limits from Experimental Data; Quantitative Aspects of Chemical and Radioisotope Carcinogenesis; Extension of Laboratory Toxicological Data to the Field of Industrial Medicine; Biological Effects of Microwave Energy; The Biological Effects of Chemical Agents; Interpretation of Toxicological Data in Animals; Toxicity of Cancer Chemotherapeutic Agents; Problems in Evaluation of the Safety of Drugs.

This is one of a series of Gordon Research Conferences which will be held on various scientific fields during the period of June 15 to September 4, 1959. These conferences are held at Colby Junior College, New Hampton School, and Kimball Union Academy, all in New Hampshire. Attendance at the Conferences is by application only. The established fee is $100 for each Conference and this covers room and board at the Conference as well as registration. Under certain circumstances a remission of a portion of the fee may be allowed. A small extra charge may be made for special accommodations. Accommodations are available for a limited number of women, and for wives or children (over 12 years) of conferees.

These conferences were established to stimulate research through an informal type of meeting consisting of scheduled lectures and discussion groups. Meetings are held morning and evening, Monday through Friday noon, and adequate time is available for informal discussion. Afternoons are free for discussion and recreation. The purpose of the program is to extend the frontiers of science through an easy exchange of ideas among scientists active and accomplished in the field. The Conferences make no publication of lectures or discussions.

Interested persons are urged to write for application forms and full information on the Conference on Toxicology and Safety Evaluations and on the subjects of other conferences. Address: W. George Parks, Director, Gorden Research Conferences, Colby Junior College, New London, New Hampshire.

Vapor Toxicity of Allyl Alcohol as Determined on Laboratory Animals*

T. R. TORKELSON, M.S., M. A. WOLF, M.S., F. OYEN, and V. K. ROWE, M.S.

Biochemical Research Laboratory, The Dow Chemical Company, Midland, Michigan

Introduction

NEW AND INCREASED uses for allyl alcohol (1-propene-3-ol; $CH_2=CH-CH_2OH$) as a material for chemical synthesis makes additional toxicological data desirable in order to make recommendations for safe handling. Data from early studies was reviewed by von Oettingen[1] in 1943. He reported that the material was readily absorbed through the gastro-intestinal tract, the lungs and the skin, leading to general irritation of most of the visceral organs and changes in blood pressure, followed by convulsions and possible death.

Smyth and Carpenter[2] have reported that allyl alcohol is high in acute oral toxicity for rats, and although it is not a very severe primary irritant of the skin and eyes of rabbits, it is readily absorbed through the skin. They also found a one-hour exposure to 1000 ppm of vapor to be lethal to rats.

McCord[3] exposed animals to concentrations of 1000, 200 and 50 ppm of allyl alcohol in air and reported the following results: 1000 ppm was lethal to rats, rabbits and monkeys after three to four hours of exposure; 200 ppm was lethal to rats and rabbits after one to 18 seven-hour exposures; and 50 ppm caused serious injury and death to rats in an average of 30 days. He reported finding widespread irritation, with pulmonary edema, hemorrhages of the gastro-intestinal tract, gastroenteritis, diarrhea and nephritis with hematuria. One of two rabbits which were exposed to 50 ppm of allyl alcohol died after 14 days, the other was sacrificed after 28 days and pathological changes similar to that seen in the rat were observed.

Recently Dunlap et al.[4] reported on rather extensive toxicological tests on allyl alcohol. The results of their acute studies are in close agreement with those published by Smyth and Carpenter.[2]

Dunlap also reported the results of repeated seven-hour exposures of groups of 10 male rats to 150, 100, 60, 40, 20, 5, 2, and 1 ppm of allyl alcohol in air for 90 day periods. The concentration of 150 ppm was reported to be highly lethal; one exposure causing several mortalities. They reported pathological changes in the lungs, liver and enteric tract. Similar signs, lesions and microscopic findings were reported in animals exposed to 100, 60, and 40 ppm but they were less severe. Except for a lack of gain in weight at 20 ppm, this and lower concentrations were reported to be without effect.

This group also described the results of sensory perception tests on humans; a concentration of 0.78 ppm was judged the lowest recognized by humans and a concentration of 25 ppm was found to cause severe eye irritation.

Kodama and Hine[5] have reported on their study of the pharmacodynamic aspects of allyl alcohol. They were particularly concerned with injury to the periportal areas of the liver which occurred in rats fed allyl alcohol and postulated a possible mode of action for this injury. Their extensive study included the effect of allyl alcohol on the following: oxygen uptake of the liver, kidney and cerebral cortex; water content of the liver; blood pressure, respiration and hemoconcentration in the dog; protection of the liver by various compounds; and dehydrogenase activity of the liver. They also described a method for the determination of micro amounts of allyl alcohol in tissue. Kodama and Hine concluded that death resulting from acute ingestion of allyl alcohol was due to cardiovascular failure rather than hepatic injury.

The studies reported herein, concerning the results of repeated exposure of laboratory animals to low concentrations of allyl alcohol in air, were conducted for the purpose of providing a basis for judging the hazard associated with repeated exposure to humans. At the time this study was nearing completion, the results of the experiments conducted by Dunlap et al.[4] appeared in print. The findings from the two studies are in agree-

* Presented at the Twentieth Annual Meeting of the American Industrial Hygiene Association, Chicago, Illinois, April 30, 1959.

TABLE I

Summary of Final Average Body and Organ Weights of Animals Receiving Repeated 7-Hour Exposures to 7 ppm of Allyl Alcohol Five Days Per Week for Five Weeks

Species	Group	Sex	No. of surviving animals	Body weight, gm	Organ weights, gm/100 gm body weight					
					Lung	Heart	Liver	Kidney	Spleen	Testes
Rat	7 ppm allyl alcohol	M	5	238	0.67	0.36	2.77	0.80	0.41	1.23
	air-exposed control	M	5	256	0.60	0.39	2.81	0.77	0.44	1.17
	7 ppm allyl alcohol	F	5	166	0.70	0.40	2.77	0.76	0.41	—
	air-exposed control	F	5	179	0.67	0.38	2.74	0.72	0.46	—
Guinea pig	7 ppm allyl alcohol	M	4	477	0.67	0.38	3.83	0.77	0.13	0.52
	air-exposed control	M	4	496	0.67	0.34	4.20	0.80	0.13	0.49
Rabbit	7 ppm allyl alcohol	F	1	2220	0.41	0.18	2.42	0.51	0.03	—
	air-exposed control	F	1	3120	0.40	0.20	3.09	0.47	0.04	—

TABLE II

Mortality and Its Cause Among Animals Receiving Repeated 7-Hour Exposures to 2 ppm Allyl Alcohol Five Days Per Week for Six Months

Species	Sex	Group	No. of animals started	No. of deaths	Reason for sacrifice or cause of death
Rat	M	2 ppm allyl alcohol	24	1	1—Found dead, cause not known
	M	air-exposed control	24	4	3—Lung infection 1—Cause of death not known
	M	unexposed control	24	3	2—Lung infection 1—Cause of death not known
	F	2 ppm allyl alcohol	24	1	1—Very severe lung infection
	F	air-exposed control	24	0	—
	F	unexposed control	24	1	1—Lung infection
Guinea pig	M	2 ppm allyl alcohol	9	0	—
	M	air-exposed control	9	0	1—Lost
	M	unexposed control	9	1	1—Found dead, cause not known
	F	2 ppm allyl alcohol	9	1	1—Found dead, cause not known
	F	air-exposed control	9	1	1—Found dead, cause not known
	F	unexposed control	9	2	2—Pneumonia
Rabbit	M	2 ppm allyl alcohol	3	2	1—Found dead, cause not known 1—Severe ear infection
	M	air-exposed control	3	2	1—Cause not known 1—Severe lung infection
	M	unexposed control	3	0	—
	F	2 ppm allyl alcohol	3	0	—
	F	air-exposed control	3	0	—
	F	unexposed control	3	0	—
Dog	M	2 ppm allyl alcohol	1	0	—
	M	air-exposed control	1	0	—
	M	unexposed control	1	0	—
	F	2 ppm allyl alcohol	1	0	—
	F	air-exposed control	1	0	—
	F	unexposed control	1		—

ment with regard to the qualitative aspects of injury resulting from over-exposure but are not in complete agreement in regard to quantitative aspects.

Experimental

PHYSICAL PROPERTIES

Allyl alcohol is a clear liquid with a characteristic pungent odor. It has a boiling point of 96.9°C and a specific gravity of 0.854 20/4°C. It is flammable (TOC flash point of 90°F) and the explosive limits are reported to be 2.5–18% by volume. One sample was used throughout this study. Vapor-phase chromatographic analysis showed the sample to be 99.5 mole per cent allyl alcohol with no impurities boiling below 150°C.

EXPERIMENTAL TECHNIQUE

The animals, equipment and procedures employed were essentially the same as described for allyl chloride in the preceding article of this journal. The same control animals served for experiments with both chemicals. A water sealed chamber similar to that described previously was used for exposure to what was calculated to be 10 ppm by volume of allyl alcohol in air. Exposures to a calculated concentration of 3.14 ppm were conducted in a vault-type chamber identical to that used for allyl chloride.

No analytical method suitable for these concentrations was available when the exposures were started but subsequent analyses using the techniques described below were found to average 6.9 ppm with a range of 6.6–7.1 ppm when the calculated concentration was 10 ppm. The analytical values for an atmosphere which was calculated to be 3.14 ppm were found to average 1.9 ppm with a range of 0.6 to 3.2 ppm. Seventy-five per cent of the values fell between 1.7 and 2.2 ppm. Analyses of the chamber air were made by drawing air at the rate of 1.0–1.2 l/min. through two fritted glass scrubbers connected in series, each containing 25 ml distilled water. The samples were transferred with repeated rinsings to 50 ml volumetric flasks and subsequently analyzed by coulometric bromination. This method was shown to give better than 90%

TABLE III

Summary of Final Average Body and Organ Weights of Animals Receiving Repeated 7-Hour Exposures to 2 ppm Allyl Alcohol Five Days Per Week for Six Months

Species	Sex	Group	No. of surviving animals	Body weight, gm	Organ weights, gm/100 gm body weight					
					Lung	Heart	Liver	Kidney	Spleen	Testes
Rat	M	2 ppm allyl alcohol	12	373	0.52	0.35	2.49	0.69	0.30	0.88
	M	air-exposed control	10	375	0.52	0.34	2.50	0.73	0.27	0.85
	M	unexposed control	12	371	0.53	0.32	2.59	0.70	0.24	0.83
	F	2 ppm allyl alcohol	11	211	0.66	0.38	2.77	0.77	0.31	—
	F	air-exposed control	12	220	0.64	0.34	2.77	0.75	0.32	—
	F	unexposed control	12	215	0.64	0.39	2.70	0.76	0.28	—
Guinea pig	M	2 ppm allyl alcohol	9	845	0.52	0.29	2.84	0.74	0.11	0.59
	M	air-exposed control	9	946	0.49	0.27	3.16	0.66	0.10	0.52
	M	unexposed control	8	933	0.55	0.26	3.31	0.70	0.11	0.53
	F	2 ppm allyl alcohol	8	867	0.49	0.30	3.87	0.71	0.13	—
	F	air-exposed control	8	875	0.67	0.29	3.64	0.77	0.12	—
	F	unexposed control	7	860	0.52	0.26	3.72	0.75	0.13	—
Rabbit	M	2 ppm allyl alcohol	1*	2,850	0.38	0.21	2.09	0.42	0.02	0.19
	M	air-exposed control	1	3,160	0.31	0.20	2.53	0.52	0.02	0.22
	M	unexposed control	3	3,200	0.35	0.29	1.77	0.47	0.02	0.22
	F	2 ppm allyl alcohol	3	3,690	0.35	0.18	2.18	0.41	0.04	—
	F	air-exposed control	3	3,570	0.43	0.23	2.05	0.46	0.04	—
	F	unexposed control	3	3,650	0.32	0.18	1.75	0.38	0.03	—
Dog	M	2 ppm allyl alcohol	1	13,300	0.80	0.80	2.47	0.46	0.17	0.16
	M	air-exposed control	1	11,300	0.86	0.98	2.65	0.44	0.25	0.19
	M	unexposed control	1	14,100	0.78	0.67	2.66	0.42	0.20	0.17
	F	2 ppm allyl alcohol	1	7,500	1.50	0.98	2.75	0.50	0.29	—
	F	air-exposed control	1	12,200	1.08	0.91	2.43	0.37	0.36	—
	F	unexposed control	1	11,000	0.76	0.81	2.31	0.41	0.21	—

* One rabbit with severe ear infection was killed two weeks before the end of the experiment and data obtained from this animal are included in the average values given.

recovery of weighed samples when the contents of the two scrubbers were combined.

Results

Seven ppm (0.016 mg/liter): The male and female rats, male guinea pigs and the female rabbit that received 28 seven-hour exposures in 35 days to a concentration of 7 ppm of allyl alcohol showed no evidence of ill-effect as judged by growth, behavior, mortality, gross appearance and final average body and organ weights (Table I). However, there was degeneration observed in the kidneys and livers of almost all animals upon microscopic examination. This was characterized in the liver by dilation of the sinusoids, cloudy swelling and focal necrosis. In the kidney the changes were similar to those commonly seen in the glomerulonephritis, necrosis of the epithelium of the convoluted tubules and proliferation of the interstitial tissue. These changes appeared to be mild in intensity and reversible.

Two ppm (0.005 mg/liter): Both sexes of rats, guinea pigs, rabbits and dogs that received 127 to 134 seven-hour exposures in 180–194 days to 2 ppm of allyl alcohol showed no evidence of ill-effect as judged by growth, behavior, mortality (Table II) and final average body and organ weights (Table III). The terminal values reported for BUN and NPN (Table IV) were in several cases below those found for the control animals for this experiment. They were however, within the normal limits for control animals from the same stock used in previous experiments and in the case of dogs are also within the normal limits listed by Dukes[7] (BUN 10–20 mg/100 ml; NPN 17–38 mg/100 ml). In the absence of pathology in the liver and kidneys which might be expected if differences in NPN and BUN were due to exposure to allyl alcohol and since the values lie within normal limits the apparent charges were not considered to be significant or

TABLE IV

Average Values for Blood-Urea-Nitrogen and Blood-Non-Protein-Nitrogen For Animals Receiving Repeated 7-Hour Exposures to 2 ppm Allyl Alcohol Five Days Per Week for Six Months

Species	Group	Sex	No. of animals	BUN mg/100 ml	NPN mg/100 ml
Rat	2 ppm allyl alcohol	F	8	15.2	46
	air-exposed control	F	8	21.4	46.5
	unexposed control	F	8	20.2	49
Guinea pig	2 ppm allyl alcohol	M	5	21.0	—
	air-exposed control	M	5	26.7	—
	unexposed control	M	4	37.7	—
	2 ppm allyl alcohol	F	5	20.7	—
	air-exposed control	F	5	28.3	—
	unexposed control	F	4	30.4	—
Rabbit	2 ppm allyl alcohol	F	3	21.6	—
	air-exposed control	F	3	21.9	—
	unexposed control	F	3	24.8	—
Dog	2 ppm allyl alcohol	M	1	14.3	32
	air-exposed control	M	1	21.2	42.5
	unexposed control	M	1	22.5	46
	2 ppm allyl alcohol	F	1	11.8	32
	air-exposed control	F	1	22.0	38
	unexposed control	F	1	21.6	Contaminated

TABLE V

Average Hematological Values for Dogs Receiving Repeated 7-Hour Exposures to 2 ppm Allyl Alcohol Five Days Per Week for Six Months

Group	When sampled	Sex	No. of animals	WBC × 10^3	Hb gm/100 ml	Hematocrit	Differential count per cent				
							Neutrophils	Lymphocytes	Monocytes	Eosinophils	Basophils
2 ppm allyl alcohol	preexposure	M	1	lost	lost	46	37	55	4	4	0
	terminal	M	1	13.8	13.5	50	54	42	1	3	0
Air-exposed control	preexposure	M	1	10.1	12.0	42	42	52	4	2	0
	terminal	M	1	10.3	14.0	51.5	48	49	1	2	0
Unexposed control	preexposure	M	1	21.4	13.0	44.5	57	42	0	1	0
	terminal	M	1	15.8	13.8	50	49	45	1	5	0
2 ppm allyl alcohol	preexposure	F	1	11.4	15	50	55	45	0	0	0
	terminal	F	1	9.8	14.5	53	63	36	0	1	0
Air-exposed control	preexposure	F	1	19.4	11.0	38	32	47	1	0	0
	terminal	F	1	14.8	14.5	49	56	38	3	3	0
Unexposed control	preexposure	F	1	22.9	14.0	45	48	46	3	3	0
	terminal	F	1	16.7	13.0	47	52	43	3	2	0

a result of the exposure to the chemical. Gross and microscopic examination of the tissues revealed no changes. The average hematological values (Table V) were within normal limits for animals of this laboratory. Examination of the rats held 60 days also showed no effect.

Human Sensory Tests: Five of the ten human volunteers reported a definite odor but no irritation when exposed to two ppm of allyl alcohol for one to three minutes.

Significance of Results

Vapor Inhalation—Repeated Exposure: Allyl alcohol has been shown to be highly toxic on repeated inhalation. Laboratory animals were slightly affected by repeated inhalation of seven ppm whereas two ppm was found to be without effect after six months of repeated daily exposure. It is of interest to note that repeated inhalation of seven ppm of allyl alcohol caused considerably less pathology than inhalation of eight ppm of allyl chloride for the same period of time. Smyth and Carpenter[2] have reported that, acutely, allyl alcohol is considerably more toxic. Neither materials caused any detectable lung injury at the levels studied as might be expected from their rather potent effect upon the nose and eyes. Injury in both cases was to the liver and kidneys. This lack of lung pathology has also been reported at 20 ppm by Dunlap *et al.*[4] The data presented herein, however, are not in complete agreement with that published by Dunlap.[4] These workers reported no effect other than slight growth depression in male rats exposed seven-hours daily to 20 ppm for three months whereas in the present study, slight, apparently reversible, pathology of the liver and kidneys was seen in rats, guinea pigs and rabbits given daily seven-hour exposures to seven ppm for one month. The data derived from the experiments described herein indicates that the highest concentration of allyl alcohol likely to be without ill-effect lies between two and seven ppm.

Industrial Hygiene Standard: On the basis of the observations reported herein, an industrial hygiene standard of five ppm is suggested for allyl alcohol. The standard as used here is defined as that concentration of allyl alcohol below which practically all fluctuations must fall during a working day where repeated, prolonged exposure to humans is likely. In view of the slight pathology occurring from exposures at seven ppm it is suggested that an overall time-weighted average for daily seven- to eight-hour exposures not exceed two ppm. Under such conditions the probability of injury would seem to be remote. It should be noted that the five ppm figure suggested here is a maximum value, not a maximum average of five ppm as suggested by the American Conference of Governmental Industrial Hygienists.

Precautions for Safe Handling

Considerable industrial experience with allyl alcohol corroborates the delayed eye irritation and muscular spasm (deep "bone ache") resulting from contact with the skin reported by Dunlap.[4] On the basis of industrial experience, previously published data and the data presented herein, the following precautions for safe handling seem appropriate.

Inhalation: The concentration of allyl alcohol in the air should be maintained below five ppm in work areas with a time weighted average for all exposures of two ppm or less. Five ppm will probably be detectable by most people but cannot be relied upon for control purposes. Suitable analytical methods must be used. Concentrations which are likely to be injurious in a short period of time will be painful to the eyes and nose. The warning odor should not be ignored.

To prevent ill-effects from inhalation, allyl alcohol must be handled in closed systems or with respiratory protection unless adequate ventilation is provided. Natural ventilation should not be depended upon for control even in outdoor areas. If spills should occur the area should be evacuated at once and re-entered only after thorough aeration. If it is necessary to re-enter before aeration is completed, adequate respiratory protection such as a full face gas mask with an organic vapor (black) canister, an air- or oxygen-supplied respirator, or a self-contained breathing apparatus should be worn.

Eye Contact: The eyes must be protected if the likelihood of contact exists. Tight fitting chemical workers' goggles or preferably a full face gas mask should be worn to prevent getting the material in the eyes.

Skin Contact: Protective clothing impermeable to the material should be worn where the likelihood of skin contact exists. Allyl alcohol should never be allowed to remain on the skin. In all cases of known contact, prompt and thorough washing is necessary. Clothing and shoes should not be allowed to become contaminated with the material and if contamination should occur, the clothing should be removed and not re-worn until thoroughly cleaned.

Ingestion: Food and drinks should not be allowed in areas where allyl alcohol is being handled. Hands should be washed with soap and water before eating or smoking.

Summary

Laboratory animals were exposed repeatedly to either seven or two ppm of allyl alcohol in air to establish conditions safe for repeated exposure. Rats, guinea pigs and rabbits had slight micropathological changes in the liver and kidneys after five weeks of repeated seven-hour exposures to seven ppm. These species and dogs received similar exposures to two ppm for six months with no detectable injury resulting.

A standard for evaluating regular seven- to eight-hour daily exposures may be defined as the concentration below which practically all analytical results must fall. The value of five ppm is considered appropriate as this standard for allyl alcohol with the proviso that the time-weighted average for all exposures not exceed two ppm.

Handling precautions for industrial usage are described.

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PROCEEDINGS OF AIR POLLUTION CONFERENCE

THE PROCEEDINGS of the National Conference on Air Pollution held in Washington, D. C., November 18–20, 1958 have been published in their entirety with a minimum of editing. An abbreviated summary of the conference's conclusions was published in the April issue of the AIHA Journal. Copies of the Proceedings (Public Health Service Publication No. 654) are not on sale at $1.75 through the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C.

Decaborane Effects upon Yeast Metabolism*

GEORGE J. LEVINSKAS, Ph.D.† and MARIAN R. PASLIAN, B.S.

Department of Occupational Health, Graduate School of Public Health, University of Pittsburgh, Pittsburgh, Pennsylvania

THE TOXICITY of decaborane ($B_{10}H_{14}$) to laboratory animals has been described in several articles. The oral and intraperitoneal LD_{50}'s for rats are 64.3 and 23.4 mg/kg, respectively. The corresponding values for mice are 40.9 and 33.2 mg/kg. Percutaneous application of dioxane-oil mixtures of decaborane to rats yielded a 48-hour LD_{50} of 740 mg/kg. Rabbits are considerably more sensitive, as evidenced by an LD_{50} of 71 mg/kg for similar treatment. Single intraperitoneal doses as low as 15.8 mg/kg were lethal to dogs.[1] When decaborane was force-fed at a level of 3 mg/kg, two of three dogs died after four doses while the third dog was sacrificed after five doses. These, and other experiments performed with repeated doses, suggested that this hydride had a cumulative effect in the animal body.[2]

Following a four-hour inhalation exposure, a 48-hour LC_{50} of 25.7 ppm was calculated for mice while rats survived exposures as high as 95.2 ppm.[3] However, a rat LC_{50} value of 46 ppm has been obtained by another investigator.[4] Exposure to concentrations as low as 4.5 ppm for a six-month period produced signs of intoxication and death in various animal species.[5]

Some of the clinical manifestations of boron hydride, including decaborane, intoxication in man have recently been reported.[6]

In 1956, the American Conference of Governmental Industrial Hygienists first proposed a tentative Threshold Limit Value of 0.05 ppm for decaborane.[7] The following year this limit was incorporated into the established Threshold Limit Values.

Attempts to study the mechanism of action of decaborane on isolated systems are confronted by the fact that this compound is sparingly soluble in water and other solvents that would not of themselves injure tissues or enzyme systems. Hill and associates had found an answer to this problem by employing yeast cells as their test system.[8] These researchers employed dilute ethanol (9.5%) as a solvent by means of which they could bring the decaborane into contact with the yeast cells. The cells were then washed free of the decaborane and resuspended in glucose at 25°C for 15 minutes. After this interval, the cells were removed and the residual glucose was determined. The decaborane was found to inhibit the utilization of the glucose by the yeast cells, the degree of inhibition being related to the initial decaborane concentration. Whether the decaborane was removed by prior washing of the yeast cells or not, the results were similar, leading the authors to interpret this as an enzyme effect.

The present report describes the use of yeast cells as a test system for further studies on the inhibitory effect of decaborane and reports the results obtained.

Methods

Fresh Fleischmann's Yeast (Standard Brands, Inc.) was washed with distilled water, centrifuged briefly at slow speeds and the supernatant liquid, containing cellular debris and the binders in the yeast cake, was discarded. This procedure was repeated three times, yielding a suspension of intact cells. The cells were "starved" by bubbling air through the yeast suspension for several hours (or overnight). The respiration of the yeast cells was then studied in a Warburg constant volume respirometer at 30°C, using conventional manometric techniques.[9] For respiration studies, each Warburg flask contained 1.0 ml of 0.1 molar phosphate buffer (pH 4.8–5.2), 10 mg of yeast cells (dry weight), and a sufficient volume of distilled water to bring the final volume up to 2.5 ml. The sidearm of the flask contained 0.5 ml of 1.5% glucose. The center well contained 0.2 ml of potassium hydroxide to absorb the liberated carbon dioxide. The fermentation studies were conducted in a similar fashion except that the potassium hydroxide was omitted and 1.0 ml of 0.1 molar bicarbonate buffer (pH 5.7–6.2) was substituted for the phosphate buffer. While the pH of the buffers varied at different times, for a given series of experiments the pH was constant. For the res-

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† Present address: American Cyanamid Company, Stamford, Connecticut.

piration studies, the flasks and manometers were gassed with oxygen while a mixture of 5% nitrogen-95% carbon dioxide was used for the fermentation studies.

For a given set of conditions, the metabolic studies were performed in duplicate or triplicate sets of flasks. In addition, manometer readings were taken at intervals of 15 to 30 minutes for two to four hours. This resulted in large numbers of individual observations. In order to assimilate this mass of data, the values for the treated cells have been expressed as percentages of their respective controls. Thus 0% represents complete inhibition and 100% reflects no effect. Each value reported represents the average of several observations made on two or three separate flasks.

Initially, the decaborane was dissolved in 10% ethyl alcohol or else prepared as an agar-saline suspension and added directly to the yeast cells in the Warburg flask. The effect was not too marked. At a later date, the decaborane was added to a suspension of yeast cells which were then incubated overnight (about 18 hours) at 30°C in a Dubnoff shaker. The next morning the cells were washed and centrifuged three times and their metabolism was studied as described above. While the direct addition of decaborane to a yeast suspension had no immediate effect upon the metabolism of yeast cells, this overnight "incubation" resulted in inhibition. This latter procedure also removed the need for a solvent to bring the decaborane into contact with the yeast cells.

Experimental Results

Decaborane, when reacted with yeast cells, inhibits both the respiration and the fermentation of the cells when glucose is employed as the substrate. The inhibition was not reversed by washing the cells with distilled water. Figure 1 illustrates the effect of decaborane upon the respiration of yeast cells and presents a dose-effect relationship such as one obtains by treating animals. Each distinct set of symbols represents a separate set of experiments.

The cross at 2.2×10^{-4} molar decaborane represents the rat intraperitoneal LD_{50} dose. This was calculated on the assumption that the rat represented an homogenous medium and that the administered dose of decaborane distributed

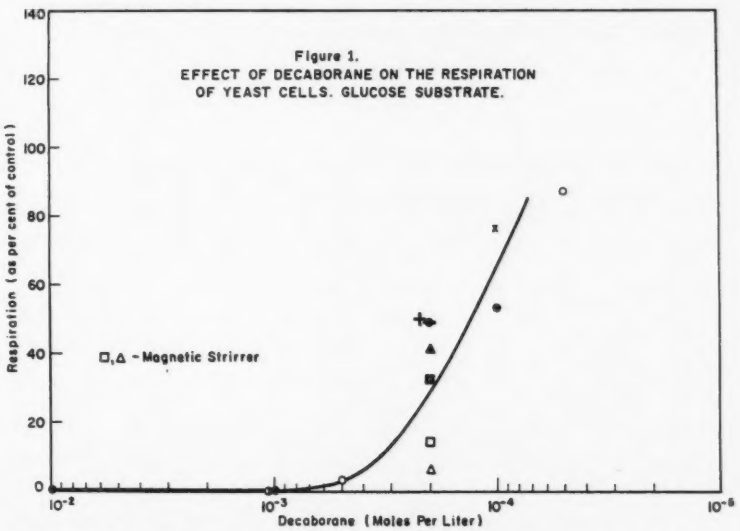

FIGURE 1. Effect of decaborane on the respiration of yeast cells (glucose substrate). The corresponding open and closed symbols at 2×10^{-4} molar decaborane concentrations represent studies performed simultaneously. The closed symbols represent data obtained by incubation in the Dubnoff shaker while the open symbols resulted from studies performed with cells agitated overnight on a magnetic stirrer. The latter are lower than the results obtained by incubation of the cells in the Dubnoff shaker where the temperature is held constant. Since the temperature of the magnetic stirrer tended to rise with time, the lessened respiration may represent a partial heat inactivation of the cells.

itself uniformly throughout the body of the rat. Thus, if the LD_{50} is expressed as 2.2×10^{-4} moles/kg, assuming that one kilogram is a liter, this would correspond to a 2.2×10^{-4} molar decaborane solution. Since this is an LD_{50} dose, the cross has been placed at the 50% ordinate. While such a calculation may not have any real significance, it was of interest to observe that the rat LD_{50} dose and the concentration which effected a 50% inhibition of the respiration of yeast cells were of essentially the same order of magnitude.

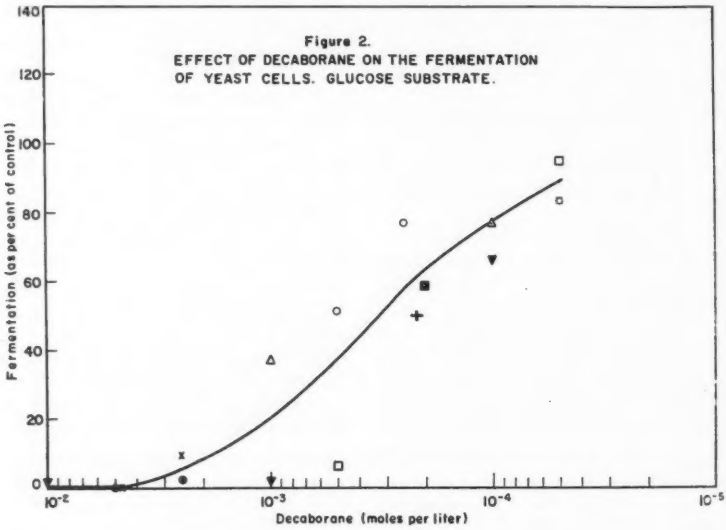
Figure 2.
EFFECT OF DECABORANE ON THE FERMENTATION
OF YEAST CELLS. GLUCOSE SUBSTRATE.
Fermentation (as per cent of control)
Decaborane (moles per liter)

FIGURE 2.

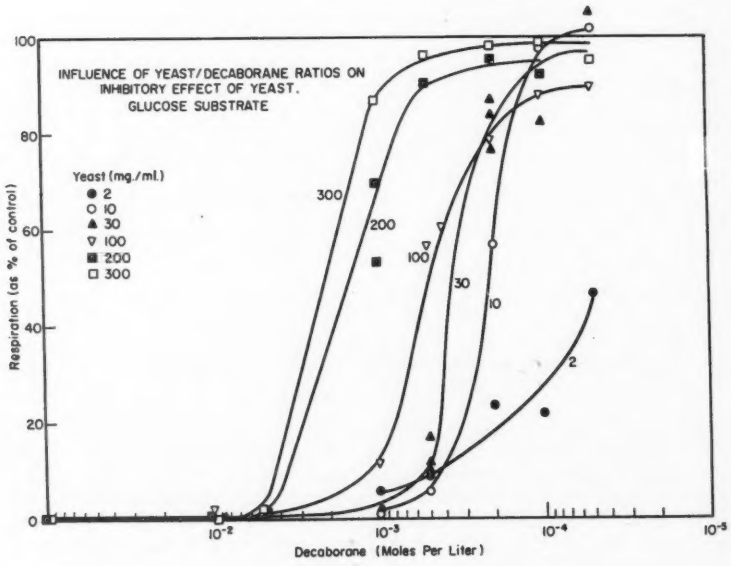

FIGURE 3.

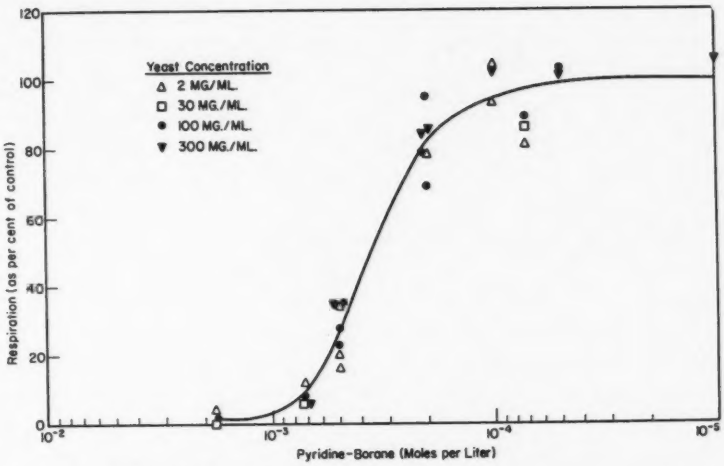

FIGURE 4. Inhibition of yeast respiration by pyridine-borane (glucose substrate)

Figure 2 presents the data obtained in fermentation studies. Thus it was possible to observe and measure the effects of this chemical on the metabolism of yeast cells.

Because of the spread in the data seen in Figure 1, an extended series of observations was made to see what other factors might be influencing the results. Different amounts of yeast, varying from 2 mg/ml to 300 mg/ml were incubated with various concentrations of decaborane and then 10 mg aliquots of the cells were taken for the respiration studies. (Approximately 10 mg. aliquots of yeast cells had been employed for the previous metabolism studies, but the yeast/decaborane ratios during the incubation period had not been closely controlled.) These additional observations are presented in Figure 3 which shows that the yeast concentration, as well as the decaborane concentration, affects the results. While yeast at a concentration of 300 mg/ml is scarcely affected by a 10^{-3} molar decaborane solution, 100 mg/ml of yeast is markedly inhibited. If the yeast concentration is reduced to 2 mg/ml, even a 5×10^{-5} molar decaborane solution exerts a 50% inhibition. The cross for the LD_{50} seen in Figure 1 would coincide with the 10 mg. line. From the data of Hill *et al.*[8], who worked with 100 mg of yeast per ml, the decaborane concentration which was 50% inhibitory was about 7×10^{-4} molar. This is in close agreement with the value of 5×10^{-4} molar obtained from our 100 mg curve.

Pyridine-borane, another boron compound whose acute toxicity has been reported,[10] is also inhibitory to the respiration of yeast cells but it does not show this dependence upon the yeast/borane ratio (Figure 4).

In the course of the uranium toxicity studies, an inhibitory effect was described which was related to the surface of the yeast cells.[11] This uranium effect is more marked than the one described with decaborane. Among the criteria considered in relating the uranium effect to the surface of the yeast cells was the promptness with which the inhibition was produced. It has not been possible to produce a similar immediate effect with decaborane, because of the insolubility of this compound.

The utilization of substrates other than glucose is inhibited by decaborane poisoned yeast cells as shown in Table I. This inhibition extends to all of the metabolites tested except ascorbic

TABLE I

Effect of Decaborane Upon Metabolism of Various Substrates

Substrate	Metabolism as % of control	
	Respiration	Fermentation
Glucose	17	53
Pyruvic acid	3	48*
Lactic acid	8	59*
Glucose	23	
Ethyl alcohol	19*	
Glycerine	40*	

* Metabolism of controls reduced in comparison with glucose substrate.

Decaborane concentration 2×10^{-4} *M* in all series
Respiration—pH 4.6 and 4.7. Fermentation—pH 6.2

TABLE II

Ascorbic Acid Metabolism of Decaborane Treated Yeast Cells

Incubation Period	Respiration Period	
	Substrate	Respiration $B_{10}H_{14}$ treated (% of control)
Decaborane	glucose	5
Decaborane and ascorbic acid	glucose	0
Decaborane	ascorbic acid	78

Decaborane concentration 5×10^{-4} molar.

Glucose and ascorbic acid present in final concentrations of 0.25%.

acid as shown in Table II. Since ascorbic acid oxidase is a copper-protein enzyme[12] while the oxidation of glucose proceeds through iron-containing enzymes, this suggests a possible reason for the different responses of these two systems. It should be noted that the prior addition of ascorbic acid to the yeast cells does not protect their glucose metabolism from inhibition.

A variety of other chemicals have been studied. Some are not metabolites for the yeast cell and therefore do not affect the respiration of the decaborane-poisoned cells. Compounds containing sulfhydryl groups, such as cysteine and glutathione do not reverse this inhibition nor do potassium ferricyanide, methylene blue or glycine.

Summary

Yeast cells, suspended in buffered solutions to which decaborane had been added, were shaken overnight in a Dubnoff shaker at 30°C. The following morning, the cells were centrifuged and the supernatant solutions were discarded. The yeast cells were then washed three times with distilled water and aliquots of the washed cells were taken for the respiration studies which were conducted with a Warburg respirometer. Buffered phosphate solutions (pH 4.8–5.2) were employed for the prior incubation and the subsequent study of respiration. In the fermentation studies, bicarbonate buffered solutions (pH 5.7–6.2) were similarly used.

Decaborane was found to be inhibitory to the respiration and the fermentation of yeast cells when glucose was employed as the substrate. In addition, the respiration of the decaborane-treated cells was markedly reduced in the presence of other substrates such as pyruvic acid, lactic acid, ethyl alcohol and glycerine. By contrast, the metabolism of ascorbic acid was not markedly affected under similar conditions.

The yeast/decaborane ratios of the incubated solutions were found to affect the results. If two different yeast concentrations were exposed to the same molarity of decaborane, the inhibitory effect was more pronounced on the dilute yeast solution. By contrast, the effect of pyridine-borane, which also inhibits the respiration, is independent of the yeast/borane ratio.

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Physiologic Study of the Summating Effects of Ethyl Alcohol and Carbon Monoxide

LOUIS J. PECORA, Ph.D.

U.S. Public Health Service, Occupational Health Field Headquarters, Cincinnati, Ohio

Introduction

THERE ARE many reports in the literature of accidental poisoning in humans, both at home and at work, stating that those who were known to have been drinking an alcoholic beverage prior to the accident, generally experienced much more severe effects than the non-users of alcohol. Recovery was usually quick and uneventful in the latter group, while in the former there were complications, slow recovery, and frequent deaths attributed to the potentiating effect of alcohol. For example, Stevens and Forster[1] reported that of thirteen individuals poisoned by carbon tetrachloride, the eleven who were known users of alcohol had much more severe reactions than did the two victims who were non-users of alcohol. Similarly, Myatt and Salmons[2] reported fifteen cases of carbon tetrachloride poisoning. Thirteen of these who used alcohol suffered severe poisoning and three of them died. Abbott and Miller[3] stated that "alcohol seemed to be a predisposing factor in eight of the ten cases" of carbon tetrachloride poisoning they observed. It has been reported[4, 5, 6] that the toxicity of calcium cyanamide in humans is greatly increased by small quantities of alcohol, even as little as that supplied by a bottle of beer.

Of the many other toxic agents that have been reported to be potentiated by alcohol, carbon monoxide has received considerable attention in the last fifteen years. During this period several reports have indicated that workers exposed to carbon monoxide lost their tolerance to alcohol and appeared to be suffering from unexplainable prolonged effects of this gas. Axelsson, et al.[7] made a pharmacodynamic study of carbon monoxide and certain anesthetics. In their report they stated that workmen exposed to carbon monoxide sometimes complained of hypersensitivity to alcohol. Tara[8] studied 28 persons suffering from chronic carbon monoxide poisoning and found that seven of these were made worse by alcohol. In this same report he stated that poisoning from mercury, lead or phosphorous was aggravated by alcoholic intemperance. Salen[9] reported on a large group of people exposed to producer gas containing 30% CO, and observed that in those who drank alcoholic beverages there was an abnormally prolonged high level of alcohol in their blood and that the people complained of lowered tolerance to alcohol. Conversely, Bjerver and Goldberg[10] in their study concluded that the alcohol tolerance of patients with chronic producer gas intoxication was no different from that in individuals not exposed to carbon monoxide.

There is no need to dwell on the pharmacologic and physiologic effects of ethyl alcohol since they are quite well known. Ethyl alcohol and carbon monoxide were considered a good combination for study since so much is known about each individually. The combination is of great interest because many people are exposed to varying levels of both substances daily while at work and in urban dwelling areas. Taxi drivers, truck drivers and garage workers form a large group that are exposed to carbon monoxide daily. Many people are also exposed to varying amounts of this gas each day through the increasingly heavy passenger car and truck traffic. The carbon monoxide concentration in some streets in our large cities may reach 0.05% which is five times the present maximum acceptable concentration.[11]

Experimental Procedure and Methods

Twelve mongrel dogs, weighing 15 to 30 pounds, were kept in our kennels about six weeks before being used in this study. During this time they were dewormed, given distemper vaccine and observed for disease conditions. Their estimated ages were from one to six years. The animals appeared to be in good health at the beginning of the experiment. The dogs were paired according to weight into control and experimental groups. Six experimental dogs, E1 to E6, were exposed to alcohol and carbon monoxide, three control dogs, C1 to C3, received alcohol alone, and three, C4 to C6, received carbon monoxide alone. All dogs were fed a conventional commercial chow. Nine were given 15% alcohol in water solution for drinking water, which they

were permitted to drink ad libitum. Immediately prior to the daily exposure to carbon monoxide in the chamber, the dogs were given about 60 ml of 15% alcohol by stomach tube and three hours later again given another 60 ml in order to assure a level of about 0.15% alcohol in the blood during the exposure to carbon monoxide. The three alcohol control dogs received the alcohol in the same manner and amount and at the same time. The blood alcohol was determined by the Newman and Newman method[12] using the Conway micro-diffusion unit. The dogs were exposed to carbon monoxide and alcohol for six hours each day, five days each week for 21 weeks. The carbon monoxide level in the atmosphere was maintained at 0.01% by volume.

The chamber used had a volume of 165 cubic feet and was air cooled to maintain a temperature of about 75°F. The relative humidity varied considerably from day to day ranging from about 35 to 65%. The air was supplied at the rate of 30 cfm. Carbon monoxide, about 97% pure, was metered through a rotameter into the air inlet at the top of the chamber to produce a concentration of 0.01%. The temperature readings and carbon monoxide readings of the chamber were made at the beginning and end of the exposure and at the second and fourth hour. The carbon monoxide concentration was checked with National Bureau of Standards indicating tubes which were standardized frequently against a Mine Safety Appliance Company carbon monoxide analyzer. The latter was calibrated with known gas mixtures assayed by the iodine pentoxide method. Since the air and carbon monoxide flows were metered accurately into the chamber, the carbon monoxide concentration could be maintained easily at a constant level.

Electrocardiograms were made every two weeks on all the dogs in a relatively quiet room. A Sanborn Visocardiette was used to record leads I, II and III. The dogs were trained to lie relaxed on their backs in a 90° wooden trough with the head on a support and with the legs loosely tied to the sides of the trough. The use of the trough insured that the dog would be in approximately the same position every time an electrocardiogram was taken, and that the dog was in an unrestrained comfortable position. It was found unnecessary to shave the hair from the legs if ample electrode paste was used. Regular electrodes used for humans were satisfactory. Rectal temperatures were obtained while the electrocardiograms were being made. After the electrocardiograms were made, 5 ml of blood was withdrawn from the jugular vein, placed in a tube with heparin and overlayed with mineral oil. This blood was later analyzed for alcohol, carbon monoxide and total hemoglobin. All the above was done within thirty minutes after removing the dogs from the CO exposure chamber.

On alternate weeks liver function tests were performed in the morning prior to exposure. Five mg bromsulphalein (BSP) per kilogram of body weight was injected intravenously and 30 minutes later venous blood was drawn, heparinized and centrifuged immediately. The dye concentration was determined according to Gaebler's method.[13] The larger dose of BSP and shorter time was used because it increased the burden on the liver and permitted a more reliable test of its functional ability. Total hemoglobin was determined by converting all the hemoglobin to cyanmethemoglobin.[14] Blood carbon monoxide content was measured by Scholander and Roughton's microgasometric method.[15]

At the end of the experiment, the dogs were sacrificed and the vital organs were examined grossly. Sections of the heart, liver, spleen, adrenals, kidneys and the lungs were preserved in Zenker's solution for microscopic study. Zenker's solution was injected into a small lobe of the lung with a syringe to insure normal fixing. These tissues were stained with hematoxylin and eosin. The whole brain was removed, sliced transversely into one-centimeter sections and placed in buffered formalin for microscopic study. Sections from the frontal, parietal and occipital lobes, the basal ganglia, thalamus and cerebellum were examined. The tissue was embedded in celloidin and 10-micron sections were cut on a frozen section microtome. Sections were stained with hematoxylin and eosin; Weigert's method for myelin and also Bodian axis cylinder stain.

Results

With the exception of control dog C3 who received only alcohol and who drank to excess, the dogs appeared to be in good health during the whole experiment. From the beginning of the experiment, dog C3 drank his alcohol copiously and ate sparingly. As a result, during the third week he developed signs of alcoholic polyneuritis that appeared to be due to a multi-vitamin deficiency. These polyneuritic signs were generalized erythematous, thickened patches of eczematous dermatitis suggestive of pellagra, plus patchy alopecia, inflamed eyelids, stomatitis, weakness, loss of equilibrium and loss of weight. His alcohol intake was then restricted to 100 ml each day and he was given two capsules of multivitamins daily for one week and one capsule daily for another week. There was an immediate marked improvement but he never regained the health status evident at the beginning of the experiment. Control dog C2 who received alcohol

only, and dog E3 who received alcohol and CO, frequently drank to excess but quickly recovered from their bouts. Dog E3 died during the tenth week from acute pneumonitis caused by accidental aspiration of alcohol. The dogs had good dispositions and were friendly throughout the whole experiment. The only neurologic abnormalities noted appeared in control dog C3 who received alcohol only.

Body temperature was relatively constant in each dog. The mean group values for body temperature were 101.1°F ± 0.23° for the alcohol controls, 101.4°F ± 0.03° for the carbon monoxide controls, and 101.3°F ± 0.12° for the alcohol-carbon monoxide group. The mean group values for body weight at the beginning of the experiment were 22.5 pounds for the alcohol controls, 22.8 pounds for the carbon monoxide controls and 22.9 pounds for the carbon monoxide-alcohol group. At the end of the experiment the mean group values were 23.8, 24.7 and 25.7 pounds respectively. Body weight increased in most dogs during the first few weeks and remained stable thereafter.

Electrocardiograms were essentially normal except for the following irregularities. At thirteen weeks alcohol control dog C3 who had normal T waves in all leads developed inverted T waves which persisted until the end of the experiment. Carbon monoxide control dog C6 who received only CO and experimental dog E4 who received both CO and alcohol, had inverted T waves in all leads at the beginning of the experiment and remained unchanged to the end. Values for the duration and amplitude of the different components fell within the normal range in agreement with normal values for dogs, published by Lannek.[16] The heart rates ranged from 70 to 100 beats per minute at the slower rates while for the faster rates in the same dog the range was from 110 to 175 beats per minute. Respiratory arrhythmia with alterations of the P and R waves, normally occurring in dogs, was observed in all the dogs and varied with the cardiac rate. It was most accentuated at low heart rates. The amplitudes of the P and R waves varied almost consistently with the respiratory arrhythmia. The range of variation of the mean electrical axis of the QRS complex in all the dogs was quite wide but still in the normal range from +30 to +80 degrees. Contrary to Lannek's report, the mean electrical axis in the individual dog was surprisingly constant and remained within a range of 10° over a period of 21 weeks. No alterations of the auriculoventricular or intraventricular conduction were noted from the electrocardiograms. Histologic examination of the heart failed to reveal any striking abnormalities. However, small degenerative lesions of individual muscle fibers were noted. The experimental group receiving both alcohol and CO showed slightly more degenerative changes in the muscle bundles of the heart than did the control groups that received CO or alcohol alone.

The bromsulphalein liver function tests were negative during the whole experiment. Serum protein and lipoprotein fractions were assayed once for all the dogs during the last week of the experiment. These determinations were made by the methods of Jencks, et al.[17, 18] Five healthy untreated dogs served as normals for comparison. The alcohol control group and carbon monoxide control group had lower than normal group mean values for the A/G ratio, 1/2.3 and 1/2.6 respectively as compared to 1/1.4 for the normal group (Table I). The group mean value for the alcohol-carbon monoxide group was 1/1.8. The total protein as determined from the areas under the electrophoretic curves was somewhat greater in all the dogs used in this experiment than in the normal group, due principally to the marked elevation of the beta fraction which was approximately double the normal values. This also accounts for the shift in the A/G ratio. The group mean total lipoprotein value for the alcohol control dogs was the same as the normal group

TABLE I

Summary of Group Mean Values of Measurements Made During the Experiment

Animal Group	No. dogs	A/G Ratio*	Total serum lipoprotein units of area*	Total Hb g/100	HbCO %	Blood alcohol ml/100	Body temperature °F
Alcohol controls	3	1/2.3	147 ± 18†	13.1 ± 0.4	—	0.14 ± .02	101.1 ± .23
Carbon monoxide controls	3	1/2.6	55 ± 8	14.8 ± 0.4	20.8 ± 2.4	—	101.4 ± .03
Alcohol-carbon monoxide	5	1/1.8	135 ± 74	14.4 ± 0.2	18.2 ± 1.3	0.11 ± .01	101.3 ± .12
Normals	5	1/1.4	148 ± 81				

* Protein and lipoprotein measurements made once on all the dogs at the end of the experiment

† Standard error of the mean, $\sqrt{\frac{\Sigma d^2}{n(n-1)}}$

value while the group mean value for the carbon monoxide control group was markedly reduced due to approximately an 80% decrease in the beta fraction. The dogs exposed to both carbon monoxide and alcohol had lipoprotein values close to the normal dogs.

Several times during the experiment determinations of serum glutamic-oxalacetic transaminase activity were made following Karmen's[19] procedure. The control and experimental animals did not exhibit any significant differences with values falling in the normal range.

Histologic study of the liver showed generally mild changes in both the alcohol control dogs and the carbon monoxide-alcohol dogs. The changes noted consisted of portal edema, dilatation of sinusoids and depletion of glycogen. Normal findings were observed in the dogs exposed to carbon monoxide alone.

Histologically the remaining organs in all the dogs other than C2 were essentially normal except for some incidental findings. Dog C2 showed chronic interstitial nephritis and triangular fibrotic subcapsular foci in both kidneys. The renal changes are thought to be unrelated to the experiment. The brain changes in these dogs were not prominent and were compatible with findings described in chronic mild anoxia. Those noted were reactive gliosis, patchy demyelination and prominence of pericellular and perivascular clear zones. Focal softening could be found sporadically but none of these lesions was large enough to be detected by the naked eye. No hemorrhage or cellular reaction were observed.

The mean group values for blood alcohol were 0.14 ± 0.02 ml/100 for the alcohol controls and 0.11 ± 0.01 ml/100 for the alcohol-carbon monoxide group, ranging from 0.05 to 0.25 ml per cent. Variations occurred from day to day in the same dog without a fixed pattern, probably due to the difference in voluntary intake of alcohol before the exposure. These analyses also indicate that the planned blood alcohol level was maintained at approximately the desired range during exposure to the carbon monoxide. The group mean values for the total blood hemoglobin content, grams per 100 ml, for the whole experiment were: 13.1 ± 0.4 for the alcohol controls, 14.8 ± 0.4 for the carbon monoxide controls, and 14.4 ± 0.2 for the alcohol-carbon monoxide experimental animals. Blood carbon monoxide content was determined nine times on all the dogs exposed to carbon monoxide during the experiment on blood drawn within thirty minutes after the end of the day's exposure. The mean group value for all the analyses of HbCO saturations for the alcohol-carbon monoxide dogs was 18.2 ± 1.3% and for the carbon monoxide controls was 20.8 ± 2.4%.

Discussion

There were noted no significant summating effects of alcohol and carbon monoxide as judged from the results of this experiment. All the dogs exposed to 0.01% carbon monoxide with and without alcohol for 20 weeks appeared to be in good health with no observable clinical signs to indicate any ill effects. The amount of alcohol given daily to each dog during the exposure is approximately equivalent to two pints of table wine or eight bottles of beer and would be comparable to what a workman could conceivably ingest during a workday. One of the alcohol control animals had signs of alcoholism, making it necessary to control his alcohol intake during the rest of the experiment. The disposition of all the dogs was friendly during the whole experiment.

Our pathologic results were in contrast to those noted by Lewey and Drabkin[20] and Ehrich, et al.[21] who observed a pathologic electrocardiogram and marked pathologic changes in dogs exposed only to CO. They noted marked degenerative changes of individual muscle fibers of the heart characteristic of anoxia in their dogs exposed acutely to high levels of CO or chronically to 0.01% CO while we noted only few small degenerative lesions of individual muscle fibers. Because of their paucity and lack of electrocardiographic changes, they were not considered important. However, had we waited three months after the exposure, as did Ehrich, et al., before sacrificing the dogs, our pathology might have been different from what we noted and might even have been similar to that observed by them. None of our dogs showed any significant abnormalities of the electrocardiogram except the one alcoholic dog who developed a negative T wave near the end of the experiment. However, there were no unusual pathologic findings in this dog's heart nor in any of the others. Liver function tests were done because of the well-known effects of prolonged alcohol ingestion on the liver and also because of strong evidence from studies with radioactive CO that large amounts of the gas are stored in the liver either because of the engorgement with radioactive HbCO or as radioactive CO combined with some other pigment as reported by Tobias, et al.[22] Tara[8] also reported that 11 of the 28 patients he studied that were exposed to CO had enlarged hard livers and that all these patients complained of decreased tolerance to alcohol. Although the above reports suggest the possibility

of liver damage, none of our tests nor histologic examinations show physiologic or anatomic liver damage of any consequence. The abnormal lipoprotein value for the CO control group suggests indirectly that there might be some altered function of the liver. Since only one assay was made at the end of the experiment and since there were no previous values for comparison, further studies are indicated in order to confirm this observation.

There are other factors such as work load that could result in chronic anoxia that could not be tested in this study but should be taken into consideration. For example, if a 15–20% HbCO saturation were attained in man while working in an environment of 0.01% CO, the maximum acceptable concentration, his arterial oxygen partial pressure would be about 45 mm Hg which is dangerously close to the steep edge of the oxygen dissociation curve. Any moderate work would increase his oxygen requirement and could bring about a lowered oxygen tension in the blood and tissues. Workers, therefore, could develop severe physiologic and anatomic changes from a constant state of anoxia. Chronic anoxia of the tissues would prevail, subsequently followed by necrosis, if of long enough duration. If a work load had been imposed on the dogs in this experiment, there might have been noticeable changes in function and anatomy of the brain and heart similar to those occurring at high carbon monoxide concentrations.

Although this experiment with CO and alcohol did not show any remarkable physiologic or anatomic changes, one should not assume that CO alone or with alcohol is safe over prolonged periods when a work load is imposed. The lower average HbCO in the alcohol-carbon monoxide dogs cannot be explained readily. This difference cannot be accounted for by changes in hemoglobin concentration since it fluctuated very little during the experiment. It is not known what influence alcohol had on the respiratory or cardiac rate during the exposure to the CO, that would alter the rate of pickup of the gas. The implication that alcohol can act as a protective agent against CO requires cautious consideration along with further detailed study to substantiate or refute it.

Summary

No significant summating effects of alcohol and carbon monoxide were noted in a physiologic study made using three groups of dogs. One group received only alcohol, another CO alone and the third both alcohol and CO. The blood alcohol level, confirmed by analysis, was kept at over .15 volume % during the daily exposure to .01% CO for six hours per day, five days per week for 21 weeks. The expected level of COHb, between 18 and 20%, after exposure to .01% CO for several hours was attained. The hemoglobin concentration was in the normal range for the three groups. The alcohol-carbon monoxide group had a mean value of 18.2% COHb while the group on CO alone had a mean value of 20.8%. The Hb mean group values were 14.4 and 14.8 g/100 respectively. The bromsulphalein liver function test was negative and the electrocardiograms were essentially normal during the whole experiment. No observable neurologic abnormalities were noted. Body temperature was normal during the whole test. Histologic study did not reveal any differences between groups nor were there any remarkable changes. A single electrophoretic assay of serum protein and lipoprotein showed a marked decrease in total lipoprotein due chiefly to reduced beta fraction.

Acknowledgments

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Course in Occupational Skin Problems

THE INSTITUTE OF INDUSTRIAL HEALTH of the University of Cincinnati announces that the fourth biennial course of instruction in *Occupational Skin Problems* will be given during the week of October 26–30, 1959. It will be presented by the Department of Preventive Medicine and Industrial Health, University of Cincinnati, in collaboration with the Occupational Health Program of the United States Public Health Service, and the Department of Dermatology of the University of Cincinnati. The objective of this course is to give physicians a greater understanding of cutaneous problems of occupational origin.

The program will be divided into daily sessions, consisting of morning lectures and clinical demonstrations, afternoon field instruction in industrial plants, and evening panel discussions. The didactic presentations will include a review of the anatomy, physiology and chemistry of the skin. Detailed consideration will be given to the etiology, diagnostic evaluation and treatment of occupational dermatoses, as well as specific measures for prevention and control of these problems. Current concepts regarding cutaneous cancer, allergic reactions and medicolegal problems will be discussed.

Physicians interested in attending the course should write for an application blank to Secretary, Institute of Industrial Health, The Kettering Laboratory, Eden and Bethesda Avenues, Cincinnati 19, Ohio. Early application is advised since attendance will be limited.

A Study of the Morin Method for the Determination of Beryllium in Air Samples

JANET WALKLEY*

Occupational Medical Service, Massachusetts Institute of Technology, Cambridge, Massachusetts

IN VIEW of the increased use of beryllium in industry and the extremely small amounts which must be determined in air and biological materials for evaluation of the environment, a rapid, sensitive method making use of readily available reagents and instrumentation is desirable.

The United States Atomic Energy Commission has recommended the following tentative levels for hygienic control:[1]

Eight-hour in-plant average concentration:	2.0 $\mu g/m^3$
Single acute instantaneous exposure:	25.0 $\mu g/m^3$
Average monthly concentration in neighborhood of plant handling beryllium compounds:	0.01 $\mu g/m^3$

The figure of 2 $\mu g/m^3$ is listed as a tentative threshold limit value by the American Conference of Governmental Industrial Hygienists,[2] and all three values are cited as tentative by the American Industrial Hygiene Association.[3] As far as is known, no standards have been established for permissible amounts in urine or other biological materials.

The use of morin as a fluorimetric analytical reagent for beryllium was first described by Zermatten.[4] It has been adapted by various workers for use with ores,[5] biological materials,[6, 7] and air samples.[8, 9, 10, 11, 12]

The proposed method using morin is similar to that used by Welford and Harley,[12] and Peterson, Fast, and Welford[13] and has been applied successfully to the determination of air samples in the author's laboratory for a number of years. The ultimate sensitivity is 0.004 μg in an aliquot (0.02 μg per sample), and the presence of relatively high concentrations of contaminants may be tolerated. It is the purpose of this paper to summarize and discuss the various factors involved and to present details of the author's method.

Instrumentation

Among commercial instruments, the Beckman DU spectrophotometer with fluorescence attachment is used by Sax and Kramlich[11] and by Sill and Willis,[14] the Beckman Photofluorometer by Klemperer and Martin,[6] the Coleman Fluorometer by Elkins,[9] and the Klett Fluorimeter by Milligan et al.[10] Other commercially available instruments with properly adapted illumination and filter systems may well be applied. A specially designed non-commercial instrument is used by Welford and Harley.[12]

In the method described in this paper, the Coleman Model 14 Spectrophotometer with fluorescence attachment is used. A UV-2 filter transmitting primarily at 436 mμ is used between the Hanovia AH-3 lamp and the sample, and a PC-2 filter between the sample and the photocell. The secondary filter transmits the fluorescent emission maximum at 550 mμ while effectively cutting out stray light from the ultraviolet lamp.

Sample Preparation

The method of treatment of air samples varies with the type of collecting medium. Analytical filter paper samples may be treated by wet ashing[10, 12, 15, 16] or low temperature ignition followed by solution of the sample.[8, 17, 18] Treatment of impinger samples is discussed by Cholak and Hubbard,[16] Elkins,[9] and Sax and Kramlich.[11] Cholak and Hubbard[16] also discuss treatment of electrostatic precipitator samples, and a combined electrostatic-filter paper method is described by Owen et al.[18]

In this laboratory, ashless filter papers (Whatman #41) and Hollingsworth and Vose H-93 fine glass fiber filters are analyzed routinely. Both are placed in 15 ml covered porcelain casseroles and ignited overnight at 500°C to destroy organic matter. Care must be taken with glass fiber filters not to overheat, as fusion may result. Mine Safety Appliances Type S unimpregnated pleated filters may be treated in the same manner, but must be placed in larger casseroles initially and transferred after ignition. Millipore filters are also used, but should not be heated directly in a muffle furnace, as complete loss of sample may result due to their sharp ignition

* Present address: Occupational Hygiene Services, 10 Post Office Square, Boston, Massachusetts.

point. They are therefore first treated with acetone, dried and slightly charred on the hot plate, then treated with concentrated HNO_3 and again dried and charred more strongly before ignition.

After removal from the muffle furnace, samples are cooled and one ml of $5N$ H_2SO_4 and 2–3 drops of concentrated HNO_3 are added. Casseroles are then covered with watch glasses and digested on the hot plate at 380°C for one hour. At the end of this time the watch glasses are removed and samples are fumed to dryness. They are then cooled and taken up with 2 to 3 ml of distilled water and two drops of $5N$ H_2SO_4. Samples are reheated to just below the boiling point and filtered through Whatman #40 filter paper into 50 ml volumetric flasks. The casserole is rubbed thoroughly with a rubber policeman and washed several times with a fine stream from a wash bottle. After dilution to the mark, samples are ready for the analytical procedure.

Reagents

(All reagents are analytical reagent grade and solutions should be filtered before use.)

Morin. As a stock solution dissolve 20 mg of pure morin in 95% ethanol and make up to 100 ml. Morin purified according to the method of Bonner may be used,[19] or a pure grade may be obtained from L. Light Co., Ltd., Poyle, Colnbrook, Bucks., England. For use, five ml of this solution is diluted to 100 ml with distilled water. Solutions are stable if refrigerated in amber bottles.

Aluminum nitrate, 0.05 molar. 18.76 grams of $Al(NO_3)_3 \cdot 9\ H_2O$ made up to one liter with distilled water.

Ammonium chloride, 25%. 25 grams NH_4Cl made up to 100 ml with distilled water.

Phenol red. Dilute 0.1 gram of the sodium salt of phenolsulfonphthalein to 250 ml with distilled water.

Ammonium hydroxide, $3N$. 20 ml concentrated NH_4OH made up to 100 ml with distilled water.

Sodium hydroxide, $4N$. 160 grams NaOH made up to one liter with distilled water.

Quinine sulfate standard. 0.4 gram quinine sulfate in one liter $0.1N$ H_2SO_4.

Potassium cyanide, 10%. 10 grams KCN made up to 100 ml with distilled water.

Beryllium standards. 0.277 gram pure BeO is dissolved in 10 ml concentrated H_2SO_4 and evaporated to dryness, redissolved in 10 ml concentrated H_2SO_4 and made up to one liter to give a solution containing 100 micrograms Be per milliliter. For working standards this is diluted 1:100 to give a concentration of one μg/ml, and two ml of this solution is diluted to one liter to give 0.002 μg/ml. Two to ten ml may be used to provide 0.004 to 0.020 μg standards.

Procedure

A 10 ml aliquot of the sample is pipetted into a 15 ml graduated centrifuge tube. One ml of $0.05M$ $Al(NO_3)_3$ is added as carrier, and one ml of 25% NH_4Cl to prevent precipitation of zinc, copper, and manganese. A drop of phenol red is added and the solution made alkaline with $3N$ NH_4OH. Samples are centrifuged for 10 minutes at 2000 rpm and the supernatant liquid discarded.

The resulting precipitate containing aluminum and beryllium hydroxides is dissolved by the addition of one ml $4N$ NaOH. When complete solution has taken place, two drops of 10% KCN are added and the sample made up to 10 ml. After recentrifuging to remove insoluble material, the sample is transferred to a cylindrical cuvette, one ml of dilute morin is added, and the fluorescence measured immediately against the quinine sulfate standard, which has been adjusted to read 100% on the drum dial. Measurements should be made rapidly as fading of fluorescence occurs.

A reagent blank and standards containing from 0.004 to 0.020 μg Be in a 10 ml aliquot may be run to obtain a standard curve. With proper adjustment of the mercury lamp and quinine sulfate concentration, results may be obtained whereby an arithmetic factor may be used to determine concentrations of unknown samples after correction of both samples and standards for reagent blank readings. We have found it necessary to run a blank and two standards with each run, since although net readings may agree between runs, variations in lamp intensity and temperature of solutions affect the absolute scale readings to some degree. For best results the mercury lamp should be allowed to warm up for at least one-half hour with the quinine sulfate standard in the chamber before readings are made.

Excellent studies of variables, confirmed in this laboratory, have been made by Laitinen and Kivalo,[20] Sill and Willis,[14] and Peterson, Fast, and Welford.[13] The two most important factors are morin concentration and NaOH normality. In the method described, the final morin concentration (1×10^{-3}%) and NaOH concentration ($0.4N$) were chosen because of the concentration range of the samples analyzed, and the linearity of response in this range. At very low NaOH and high morin concentrations, the fluorescence due to reagents is excessive and other metals begin to interfere, and at high NaOH and low morin con-

centrations, the fluorescence due to the beryllium becomes negligible in comparison with the blank. With other instruments it will probably be found necessary to vary these factors as well as the quinine sulfate standard concentration.

Insignificant differences were found when other reagents (aluminum nitrate, ammonium chloride, potassium cyanide) were varied within plus or minus 25% of the recommended amounts. Studies were not done on the effect of total salt concentration, since foreign ions are effectively removed during initial precipitation and recentrifugation of the sample.

Unsatisfactory results were obtained in this laboratory with all reducing reagents tested for control of fluorescence fading. With good technique it has been found possible to measure accurately the fluorescence of samples immediately after the addition of the morin. Greater sensitivity is obtained in this way, since if samples are measured after a time lapse to permit equilibration of fluorescence, the difference between blank and samples is materially decreased.

Study of Interferences

According to Sandell[6] morin becomes nearly a specific reagent for beryllium in normal NaOH solution, although zinc, lithium, and calcium may still fluoresce. Small amounts of copper, silver, gold, manganate, and chromate reduce the fluorescence by oxidation of morin. In the present method it was assumed that the initial NH_4OH precipitation would effectively separate interfering amounts of metals soluble in ammonium salts, and that the addition of KCN would prevent interference from residual zinc and copper. In order to confirm this, tests were made of known and suspected interferences, as well as of a number of ions which might be associated with beryllium in plant processes. Known amounts of these ions were added to standard aliquots containing 0.0, 0.01, and 0.02 μg of beryllium. Untreated standards were run with each set of "spikes" to eliminate the effects of instrument and reagent variables. One hundred μg of the ions under test were added to give a concentration factor of 1:10,000 in an aliquot containing 0.01 μg of Be.

The most serious interferences at these levels appear to be iron and zinc. If iron is not recentrifuged out of solution after dissolving the sample in NaOH, the net fluorescence is equal to a loss of 0.01 μg Be. If $Fe(OH)_3$ is removed from solution by recentrifuging, however, this effect is overcome. At much higher levels of iron some loss of beryllium may be expected by occlusion in the ferric hydroxide precipitate, and it may be necessary to add complexing agents to prevent this type of interference.

TABLE I

Effect of Foreign Contaminants on Beryllium Recovery

Contaminant	Amount (μg)	Be equivalent (μg)*
Ca	100	none
Cu	100	none
F	100	none
Fe	100	none
Mg	100	none
PO_4	300	none
Ti	100	none
U	100	none
Zn	100	0.004
Zn	75	none
Zr	100	none

* Sensitivity = 0.004 μg Be per aliquot.

At the 100 μg level, zinc increases the blank reading by the equivalent of 0.004 μg of Be. This effect is not noticeable at a level of 75 μg of Zn. With large amounts of zinc, it would be advisable to dissolve the initial NH_4OH precipitate in acid, add one ml of NH_4Cl, and reprecipitate with NH_4OH.

Recovery of Beryllium from Air Samples

A series of "spiked" samples were prepared on the three types of collecting media commonly used in this laboratory. Beryllium was added as the sulfate, prepared according to the directions given in this paper, and as the oxide, prepared from a suspension of minus 320 mesh fused (refractory grade) BeO in tetrabromoethane. Preparation of spikes at low levels is difficult, particularly with a BeO suspension where slight variation in particle size may affect the results significantly, however results indicate that this method is very satisfactory. Samples were pre-

TABLE II

Recovery of Beryllium from Spiked Samples

Be added (μg)	Medium	Chemical Form	Be Recovered (μg)*
0.05	Millipore	BeO	0.08
0.63	"	"	0.61
0.15	"	$BeSO_4$	0.23
0.50	"	"	0.45
0.63	Glass Filter	BeO	0.48
6.3	"	"	6.8
0.10	"	$BeSO_4$	0.12
1.0	"	"	0.88
12.6	Whatman 41	$BeSO_4$	12.6
0.63	"	"	0.64

* Sensitivity = 0.02 μg Be per sample.

TABLE III
Results of Typical Field Samples

Sample description	Sampling medium	Air flow	Time	Total μg Be	μg Be/m³
1. Blank	Whatman #41	—	—	0.03	—
2. Background, machine shop	"	16 cfm	10 min	0.54	0.12
3. Turning compacted and sintered Be cylinder, using flexible hose exhaust	"	"	24 "	19.3	1.8
4. Turning Be metal cylinder, using vacuum exhaust	"	"	12.5 min	0.29	0.51
5. Turning 2% Be-Cu cylinder without ventilation	"	"	5 "	0.70	0.31
6. Boring and facing Be metal cylinder using vacuum exhaust	"	"	15.5 "	0.55	0.78
7. Blank	Glass Fiber	—	—	<0.02	"
8. Background, machine shop, station A	"	2.75 lpm	93.5 hrs	1.0	0.065
9. Same, station B	"	2.50 "	"	7.0	0.50
10. Same, station C	"	2.60 "	49.0 "	7.0	0.91
11. Blank	Millipore	—	—	<0.02	—
12. Back ground, machine shop, station A	"	1.47 lpm	44.0 hrs	0.20	0.05
13. Same, station B	"	1.79 "	"	0.28	0.06
14. Same, station C	"	1.59 "	"	0.32	0.08

pared and analyzed according to the method given in this paper, and results are presented in Table II. Blanks on the three types of media are usually below the limit of detection, but may run up to 0.05 μg of Be. Some of the discrepancies noted may be attributable in part to spike preparation itself, and it is obvious that accuracy is inevitably reduced with increased manipulation of the samples. In air sampling procedures, however, these factors are outweighed by errors introduced before the sample arrives at the laboratory.

Table III illustrates results obtained on typical field samples using the three types of media used in the spiked samples.

Summary

A method is presented for the fluorimetric determination of beryllium with morin which is sensitive to 0.004 μg of Be in an aliquot or 0.02 μg per sample. With 0.01 μg Be in an aliquot there is no interference from 100 μg of Ca, Cu, F, Fe, Mg, PO_4, Ti, U, and Zr. At a concentration of 100 μg, Zn gives a fluorescence equivalent to 0.004 μg of Be, but does not interfere at a level of 75 μg. The method has been successfully applied to air samples on ashless filter paper, glass fiber filters, M.S.A. type S pleated filters, and molecular type filters. Directions are given for the preparation of these samples. Recoveries of Be from spiked samples show that the method is satisfactory for the tolerances and ranges encountered in air sampling procedures, and results of typical field samples are shown.

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Air Pollution Instruments

PUBLICATION of the third edition of *Instruments for the Study of Atmospheric Pollution* has been announced by The American Society of Mechanical Engineers. The booklet, which is a comprehensive listing of devices useful in air pollution investigation and control, has been completely revised and enlarged, according to F. S. Mallette, executive secretary of the ASME Committee on Air Pollution Controls. The committee sponsored compilation of the work.

The booklet is designed to aid businesses and government in obtaining suitable equipment for the study of their particular smog problems. Categories include measuring of smoke density samples, particles, gases, vapors and liquids, wind, rain, snow and humidity, all either separately or together. All individual listings, which range from filter paper samplers to gas chromatographic equipment plus the names and addresses of their manufacturers, have been brought up to date.

The format of the second edition, which was an innovation, has been kept. A ten-part outline lists the instruments under general headings. The second section amplifies the outline by a complete breakdown of categories. All of these are keyed to the manufacturers' names and adresses, which comprise the third section. The booklet, which is priced at $2.00 per copy, is available from the Order Department, ASME, 29 West 39th Street, New York 18, N. Y.

Radiation Protection Laws and Codes– A Scramble for Action

W. A. McADAMS

General Electric Company, Schenectady, New York

ONE OF THE most troublesome problems of the expanding atomic energy industry is the matter of regulatory control by government, at all levels. Until 1954, the atomic energy industry was a tight government monopoly owned and operated by the Federal Government through the Atomic Energy Commission. The situation was radically changed when Congress passed the Atomic Energy Act of 1954. This new legislation permitted industry, educational institutions, medical and research organizations, and other groups to obtain and use nuclear fuels and by-product materials in ways not completely under control of the AEC. As soon as the Act was passed it was realized that the laws and codes of the various states would have to be reexamined to determine if they were adequate to meet the problems of the nuclear age.

Almost immediately the states began to make studies of their laws and codes and as a result it is estimated that since 1954, more than 250 legislative and other regulatory proposals dealing with atomic energy have been considered by the state governments. About 100 of these proposals have already been approved.

Radiation Protection Codes

If one examines these many pieces of legislation, it is immediately apparent that the radiation hazards associated with the use of atomic energy are of more concern to more people than any other aspect of the industry. This concern cannot be completely justified, for the atomic industry still contributes only a very small fraction of the total radiation received by either the workers in the field or the general public. It is true, however, that in the last few years thousands of installations have begun to use radioactive materials for the first time, and legislators and agency officials are duty-bound to take cognizance of this fact. It turns out that when government organizations begin to study the matter, they become aware of other radiation problems which have needed solution for some time.

At the beginning of 1955, there were few radiation protection laws and codes in effect, and of these, only a few had been written specifically for the atomic industry. At the federal level, in addition to a few licensing procedures adopted by the AEC, the U.S. Public Health Service, The Bureau of Foreign Commerce, the ICC, the Food and Drug Administration, the Labor Department, and at least six other departments or agencies of the federal government had some radiation protection regulations in effect. Without exception, these regulations, some in revised form, are still in effect. In the meantime, the AEC has promulgated a number of additional rules, but these apply to only the small fraction of sources which are licensed by the AEC.

At the state level at the beginning of 1955 only California had a detailed radiation protection code in effect. Several other states, including New Jersey, Florida, Oregon, Colorado and others had a few general rules for radiation protection as a part of their occupational health codes. Two states, Wyoming and Delaware, required a permit to own or use a radiation source, and in Delaware this rule applied to all sources. Nine states had rules for the use of shoe-fitting fluoroscopes.

During 1955, the New York State Departments of Health and Labor each developed detailed radiation protection codes, following closely the advice of the National Committee on Radiation Protection and Measurements which was in the process of writing recommendations for such regulations. Both codes were put into effect by the end of 1955. The recommendations of the National Committee were published in December 1955.

During 1956, the Texas and Pennsylvania Departments of Health adopted, as a code, the regulations suggested by the National Committee. In addition, the Kentucky Department of Health revised its occupational health rules to make all provisions of them apply to radiation and radioactive materials.

In 1957, Massachusetts and Connecticut adopted radiation protection rules similar to those recommended by the NCRPM, and Michigan adopted a very complicated code which included most of the AEC radiation protection

regulations in addition to those recommended by the NCRPM. Alaska, North Dakota, South Dakota and Illinois adopted registration procedures for sources.

In 1958, Minnesota promulgated a detailed radiation protection code, the first one with provisions for the design, construction and operation of nuclear reactors. Colorado, Kansas and New Jersey took steps to provide for the registration of sources, and several other states developed draft proposals for either complete codes or for a registration procedure or both. Thus, by December 24, 1958, eight states had approved strong radiation protection codes, and 16 states had provided for the licensing or registration of sources (including seven of the states with strong codes). Eleven states had adopted enabling acts on radiation protection, all delegating the control, or part of it, to the Department of Health. Altogether, nearly two thirds of the states had some general radiation protection rules and about 20 had some special regulations for the control of shoe-fitting fluoroscopes. Nearly all of the radiation protection rules approved by the states to date have followed closely the recommendations of the National Committee on Radiation Protection and Measurements.

In addition to these many regulations in the specific area of radiation protection, many others dealing with the broad problem of atomic energy are of interest here. For example, eleven states have passed acts for co-ordinating atomic development activities within the state, and at least 32 states have established atomic energy study committees. The current regulatory picture for the 49 states is summarized in Table I.

TABLE I

States with strong radiation protection code		
California	Michigan	Pennsylvania
Connecticut	Minnesota	Texas
Massachusetts	New York	
States with registration procedure for sources		
Alaska	Massachusetts	Pennsylvania
Colorado	Michigan	South Dakota
Connecticut	Minnesota	Texas
Delaware	New Jersey	Wyoming
Illinois	New York	
Kansas	North Dakota	
States with some general radiation protection rules (in addition to states listed above)		
Arkansas	*Mississippi	*Utah
Florida	*Montana	*Vermont
*Georgia	*New Mexico	*West Virginia
Indiana	Ohio	Wisconsin
Kentucky	Oregon	
	Tennessee	
States with act for Health Department control of radiation		
†California	Massachusetts	North Dakota
Colorado	†New York	Oregon
Connecticut	New Jersey	South Dakota
Illinois		†Wisconsin
States with atomic energy study committees		
Alabama	Kentucky	Ohio
Arkansas	Louisiana	Oklahoma
California	Maryland	Oregon
Connecticut	Massachusetts	Pennsylvania
Colorado	Michigan	Rhode Island
Delaware	Minnesota	South Carolina
Florida	Mississippi	Tennessee
Georgia	Nevada	Texas
Illinois	New Jersey	Virginia
Iowa	New York	Washington
Kansas	North Carolina	Wisconsin

* Rules apply to only part of sources in the state.
† Limited in scope.

Zoning and Building Construction Codes

Many states now have agencies which develop and administer zoning and building construction regulations. A number of governmental officials within these states believe it is necessary to change these codes to make them apply specifically to radiation facilities. A few states have already modified existing rules to cover nuclear reactors and radiation generating machines. A typical example of the kind of proposal being suggested is a bill considered last year by the Arizona legislature. The bill (which did not get out of committee) would have prohibited the construction of any reactor or missile plant within 60 miles of a city of more than 100,000 population, or within 40 miles of a city of more than 10,000. If such a regulation were allowed to set a precedent, the whole future of atomic energy and radiation applications would be seriously jeopardized. If all the states passed such a bill, the construction of reactors and missile plants would be effectively outlawed in 30 or more states. It is obvious that such proposals must be given careful study, not only by the lawmakers, but by industry, labor and other groups as well.

Recommended Action at the State Level

It seems clear from these many regulatory proposals that the states are convinced they must take some regulatory action in the radiation protection field. If this trend continues—and it almost certainly will—the states must be extremely cautious about the laws and codes they

adopt, or the development of peaceful uses of radiation and atomic energy will be severly handicapped. It is difficult to recommend a general policy for radiation protection control which will serve all the needs of all the states, but the following general principles should be applicable to most situations:

1. Before passing any new law or promulgating any new regulations, the state should examine the laws and codes already in effect. In many cases simple word changes to cover radiation and/or radioactive materials will be all the action that is necessary for the time being.

2. The state should develop its rules carefully, preferably in a step-by-step process, and should be prepared to enforce each rule adopted. A simple registration procedure for all sources to identify the extent of the problem is a logical first step. Simple enabling legislation to assign responsibility for control is probably the next step needed. Additional rules can be developed as quickly as they can be enforced.

3. Rules adopted should be clear, but not too detailed or specific. Exposure limits should be flexible and whenever possible, methods of controlling exposure should be left to the management of the separate installations.

4. The regulations of other states should be examined and desirable features included in the interest of uniformity. Some typical points where uniformity can and should be achieved are the following: definitions of basic terminology, exemptions, exposure limits, warning signs and symbols, and the recording of exposures received.

5. The recommendations of the National Committee on Radiation Protection contained in NBS Handbook 61 (now under revision), should be followed to the maximum extent.

Conclusion

The states have made it clear that they consider radiation protection an inherent responsibility of their agencies which normally protect the health and safety of the people. So far, the actions taken by the states have been well-considered and the codes adopted have been surprisingly uniform. However, the states must be extremely cautious about new proposals for control of radiation hazards, and must make sure the regulations adopted are sound, uniformly simple, and not overly restrictive.

Electronic Calibration Center

THE ELECTRONIC CALIBRATION CENTER of the National Bureau of Standards was formally dedicated at the Bureau's Boulder (Colorado) Laboratories in August, 1958. This Center will provide government, industry, research centers, and the military services with access to the nation's primary electronic standards. In a new wing of the Radio Standards Laboratory, personnel of the new Center will calibrate the master working standards which in turn are used to calibrate the instruments on production lines and in research laboratories. On a fee basis instruments will be calibrated for such organizations. Persons desiring to utilize these services should write to the U.S. Bureau of Standards, Electronic Calibration Center, Boulder, Colorado.

Research Highlights

RESEARCH HIGHLIGHTS of the National Bureau of Standards, Annual Report, 1958, has been issued as NBS Miscellaneous Publication No. 226. This 138 page report describes a wide range of scientific studies, laboratory experiments, instrument developments and technical publications. Such items as gage blocks, heat resistant coatings, metals and alloys, telemetering equipment, infrared measuring methods and equipment, electronic standards, pyrometer calibration, an atom beam wavelength source, and numerous other technical areas are discussed. The NBS's role in the International Geophysical Year and the co-operative programs in science and industry are described.

The report may be ordered from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C., for 45 cents.

HYGIENIC GUIDE SERIES

Propylene Oxide

I. Hygienic Standards

A. RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours): 150 parts of vapor per million parts of air, by volume (ppm).[1]

(1) *Basis for Recommendation:* Toxicological observations on animals.[3]

B. SEVERITY OF HAZARDS:

(1) *Health:* Moderate, for both acute and chronic exposures. Exposed animals exhibited eye irritation, nasal irritation, difficulty in breathing, drowsiness, and occasionally, incoordination. The symptomatology was determined by the total dose. Temporary weight loses of survivors disappeared within fourteen days. Repeated exposures to 457 ppm depressed animal growth and pulmonary changes were found microscopically. Aqueous solutions confined to the skin can cause severe skin damage.

(2) *Fire:* High. Explosive limits in air at 760 mm are given as 2.1% to 21.5% by volume,[1] although more recent observations[4] indicate the upper level to be 38.5%. Flash point: −35°C (open cup).[1]

C. SHORT EXPOSURE TOLERANCE: Inhalation of 4,000 ppm for four hours has been reported as being lethal to rats.[5] Concentrations of 4,000 ppm for one-half hour, 2,000 ppm for two hours, and 1,000 ppm for four hours failed to cause organic injury in rats.[3] Another author reports the LC_{50} for rats as being 4,000 ppm for four hours.[2]

D. ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE: Unknown.

II. Significant Properties

Chemical formula:	$CH_3—CH·CH_3$
Molecular weight:	58.08
Specific gravity:	0.8394 (20°/20°C)
Boiling point:	33.9°C at 760 mm
Freezing point:	−104.4°C
Vapor pressure:	445 mm. at 20°C
Solubility:	40.5% by weight in water at 20°C; completely in alcohol and ether
Odor threshold:	200 ppm[2]
At 25°C and 760 mm Hg,	
1 ppm of vapor:	0.0024 mg/liter
1 mg/liter:	416 ppm

III. Industrial Hygiene Practice

A. RECOGNITION: May be recognized by its ethereal odor and irritant effects. Propylene oxide is used as a chemical intermediate in making various oil additives, antioxidants, glycols, and polyglycols. It can be used directly as a solvent and sterilizing agent.

B. EVALUATION OF EXPOSURES:

(1) *Direct instrumentation:* None available.

(2) *Chemical:* Same as for ethylene oxide. Collection in an all-glass scrubber, containing a solution of sulfuric acid and magnesium bromide, with subsequent titration of the excess acid by standard sodium hydroxide.[3]

C. RECOMMENDED CONTROL PROCEDURES: Enclosure of processes, accompanied by

local or general ventilation. Eye protection should be worn and personnel instructed in safe handling procedures.

IV. Specific Procedures

A. First aid: Clothing and shoes contaminated with proplyene oxide should be removed and the skin thoroughly washed. In case of eye contact, irrigate immediately with large quantities of water for at least 15 minutes.

V. References

1. Curme, O. O., and F. Johnston, Editors: *Glycols.* Reinhold Publishing Corp., New York (1952).
2. Jacobson, K. H., et al.: *AMA Arch. Ind. Health, 13:* 237 (1956).
3. Rowe, V. K., et al.: *AMA Arch. Ind. Health, 13:* 228 (1956).
4. Rowe, V. K., (Dow Chemical Co.): Personal communication.
5. Smyth, H. F., Jr. and C. P. Carpenter: *J. Ind. Hyg. & Tox. 30:* 63 (1948).

Tetrahydrofuran

(Tetramethlyene Oxide)

. Hygienic Standards

A. Recommended maximum atmospheric concentration (8 hours): 200 parts of vapor per million parts of air, by volume (ppm).[1]
   (1) *Basis for Recommendation:* Animal experiments.

B. Severity of hazards:
   (1) *Health:* Moderate, for both acute and chronic exposures. Over-exposure may result in nausea, dizziness and headache, but these symptoms disappear rapidly on access to fresh air. Early studies which showed kidney effects were probably due in part, at least, to use of impure material.
   (2) *Fire:* High. Explosive limits in air at 760 mm are 2.0% and 11.8% by volume. Flash point: −14.5°C.
   Peroxides are likely to form on storage and may decompose violently if the solvent is concentrated by evaporation.

C. Short exposure tolerance: 25,000 ppm will cause anesthesia. Concentrations of 17,000 ppm appear to be safe for three hours.[3]

D. Atmospheric concentration immediately hazardous to life: Unknown.

II. Significant Properties

Chemical formula:	$(C_2H_4)_2O$
Molecular weight:	72.10
Specific gravity:	0.888
Boiling point:	66°C
Freezing point:	−108°C
Vapor pressure:	142 mm at 20°C
Solubility:	Miscible with water and most organic solvents
At 25°C and 760 mm Hg,	
1 ppm of vapor:	0.0029 mg/liter
1 mg/liter:	240 ppm

III. Industrial Hygiene Practice

A. Recognition: By its ethereal odor. It is used as a solvent for resins and plastics and for chemical reactions.

B. Evaluation of exposures:
   (1) *Direct instrumentation:* Combustible gas indicator can be used.
   (2) *Chemical:* Absorb in cold water and determine colorimetrically by reaction with phenanthraquinone in sulfuric acid.[2]

C. Recommended control procedures: Provide sufficient ventilation to maintain atmospheric concentration below 200 ppm. Eye protection is advisable when handling liquid solvent. To retard peroxide formation avoid exposure to light.

IV. Specific Procedures

A. First aid: In case of eye contact, irrigate immediately with large quantities of water for at least 15 minutes.

B. Specific medical procedures: Treatment of exposure is non-specific.

V. References

1. American Conference of Governmental Industrial Hygienists: *AMA Arch. Ind. Health, 18:* 178 (1958).
2. E. I. DuPont de Nemours and Company: Electrochemicals Department, Physiological Properties of Tetrahydrofuran. Wilmington, Del. (1947).
3. Hofmann, ——, and Oettel, H.: *Archiv. Fur Exper. Pathologie U. Pharmakologie 222:* 223 (1954).

Polonium^{210}

I. Hygienic Standards

A. Recommended maximum atmospheric concentration (8 hours):

Authority	Microcuries per milliliter of air (μc/ml)	
	Soluble compounds	Insoluble compounds
NCRP[7]	6×10^{-10}	2.1×10^{-10}
CFR Part 20[1]	6×10^{-10}	2×10^{-10}
ICRP[3]	1.5×10^{-9}	3×10^{-10}

(1) *Basis for Recommendation:* Calculations based on a maximum irradiation level of 300 mrem/week to the critical organ. For soluble compounds the critical organ is the spleen[2] with the effective half life being 40 days.[8] For insoluble compounds, the critical organ is the lung[4] with the effective half life being 31 days.[4]

B. Severity of hazards:

(1) *Health:* Severe for both chronic and acute exposures. Because of its very high specific alpha activity, an extremely small amount of polonium^{210} gives a high internal radiation dose. For example, a single spherical particle one micron in diameter has an activity of 0.02 μc, which is the permissible body burden for insoluble polonium according to ICRP, and three times the permissible body burden according to NCRP.

C. Short exposure tolerance: Calculations of a permissible brief exposure air concentration based on a permissible polonium^{210} burden in the critical organ can be made using the same parameters employed in the calculation of the 8-hour threshold limit values. For a single ½-hour exposure the calculated permissible levels are:

	NCRP and CFR Part 20, μc/ml	ICRP, μc/ml
Soluble compounds	7.2×0^{-7}	1.4×10^{-6}
Insoluble compounds	1.4×10^{-7}	4.0×10^{-7}

D. Atmospheric concentrations immediately hazardous to life: Unknown for man. For cats, dogs, rabbits, and mice the LD_{50} at 20 days is about 80 μc/kg. For rats, the most sensitive species tested, the 20 day LD_{50} was about 40 μc/kg. The effect which was the most sensitive criterion of polonium injury was the shortening of life span. The lowest single dose of polonium causing a measurable shortening of life span was 0.5 μc/kg of body weight, equivalent to 35 μc in a 70 kg (155 lb) man.[6]

II. Significant Properties

A silvery grey, soft metal much like lead in appearance. Decay product (radium F) of the uranium-radium chain (see Hygienic Guide: Radon and Its Daughters). It is an alpha emitting element decaying directly to a stable isotope, Pb^{206}. About 0.001% decays through an excited state of Pb^{206} which results in very small amounts of x- and gamma radiation in addition to the predominant alpha.

Chemical symbol:	Po
Molecular weight:	210
Specific gravity:	9.2–9.4
Melting point:	246–252°C
Vapor Pressure:	$\log P = (-5380/T) + 7.23$ T = °K, and P = mm of Hg
Radiation emitted:	essentially all alpha
Radioactive half-life:	138.4 days

Specific gravity:	4.50×10^5 curies/gram
Alpha energy:	5.30 Mev

III. Industrial Hygiene Practice

A. RECOGNITION: Polonium is useful only as an alpha emitter. It is used in static eliminators and alpha sources. When combined with beryllium it becomes a neutron source. An interesting application is the ionization anemometer for measuring very low air flow rates.[5] In it, the fraction of the alpha radiation emitted by a polonium source which reaches the detector is proportional to the air flow rate.

B. EVALUATION OF EXPOSURES: Collection of dust on filter paper followed by analysis by alpha counting, when appropriate corrections are applied for imbedding and self-absorption. The polonium may be removed by leaching with acid and allowed to plate spontaneously on a nickel or copper disc, followed by alpha counting.

C. RECOMMENDED CONTROL PROCEDURES: Extreme care must be exercised in handling to prevent inhalation or ingestion. There is no external radiation hazard. When it is used for its alpha emitting property only, it is adequately shielded if covered by a thin metallic film. Processing is performed in glove box hoods under negative pressure.[6] In all forms it is said to migrate through areas of use and out of containers[5] in amounts proportional to source strength, because of its appreciable vapor pressure.

IV. Specific Procedures

A. FIRST AID: Remove exposed person from contaminated area; remove contaminated clothing and wash thoroughly. Collect all urine and fecal specimens to help determine extent of Po intake. Intramuscular BAL was found to be effective in reducing the toxicity in animals of Po introduced intravenously, but not effective for Po administered orally.[9]

B. SPECIAL MEDICAL PROCEDURES: Urinalysis is a useful personnel monitoring device since the maximum excretion in the urine occurs within 24 hours of the exposure. Polonium in urine can easily be chemically plated onto a disc, and counted for alpha activity. The Mound Laboratory[6] restricts the activity of personnel whose urines have an alpha activity greater than 24 d/min per 50 ml (2×10^{-7} mc/ml).

V. References

1. Federal Register: *Standards for Protection Against Radiation. Title 10 Code, Part 20*, Supt. of Documents, Washington, D.C. (Jan. 1957).
2. FINK, R. M.: *Biological Studies with Polonium, Radium, and Plutonium, N.N.E.S. Div. VI*, Vol. 3, McGraw-Hill, New York (1950).
3. ICRP: *Recommendations of the International Commission on Radiological Protection, Brit. J. of Radiology, Supplement #6* (Dec. 1954).
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5. LOVELOCK, J. E. AND E. M. WASELEWSKA: *J. Sci. Inst. 26:* 367 (1949).
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9. STANNARD, J. N. AND H. HAYDON: *The Effect of BAL on Distribution and Excretion of Orally Administered Polonium*[210], *Univ. of Rochester Report UR-429*. Supt. of Documents, Washington, D.C. (1956).

Radon and Its Daughters

I. Hygienic Standards

A. RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours):

Authority[a]	$\mu\mu c/l$[b]	d/m/M³[c]	$\mu g/M^3$[d]
NBS[11]	30	6.6×10^4	2×10^{-7}
CFR Part 20[2]			
Adults	100	2.2×10^5	7×10^{-7}
Minors	3.3	7.3×10^3	2×10^{-8}
ICRP[3]	300	6.6×10^5	2×10^{-6}

Note:
(a) NBS: National Bureau of Standards
CFR Part 20: Code of Federal Register, Part 20
ICRP: International Commission for Radiation Protection.
(b) $\mu\mu c/l$: Micro microcuries per liter of air.
(c) d/m/M³: Disintegrations per minute per cubic meter of air.
(d) $\mu g/M^3$: Micrograms per cubic meter of air.

(1) *Basis for Recommendation:* A marked increase in bronchial carcinoma was reported among European uranium miners during the first three decades of this century.[13] The radon levels in these mines have been estimated to have averaged about 3000 $\mu\mu c/l$ in the working areas. Largely on the basis of this experience, Evans[4] recommended a maximum permissible occupational exposure of 10 $\mu\mu c/l$. This was later reinterpreted by the NCRP which proposed that the concentration of radon plus all its daughters not exceed 30 $\mu\mu c/l$ for a 40-hour week (10 $\mu\mu c/l$ for 168 hours). On the basis of the same background experience, Holaday[16] has proposed 100 $\mu\mu c/l$ as a maximum permissible concentration for each of the daughters, disregarding the radon.

B. SEVERITY OF HAZARDS:

(1) *Health:* Extra hazardous for both acute and chronic exposures. Lung cancer was reported to account for between 50% and 80% of all deaths among uranium miners of Czechoslovakia and East Germany.[9] There is considerable evidence that this resulted, at least in part, from radon exposure, although the precise extent of those exposures cannot be established with a high degree of reliability. Because of the long latent period, no comparable experience has been derived on American miners.

C. SHORT EXPOSURE TOLERANCE: A one-hour exposure to 7.5×10^9 $\mu\mu c/l$ has been measured as an LD_{50} for mice.[10]

D. ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE: Unknown.

II. Significant Properties

Radon is a noble gas and a decay product of radium, which in turn is a decay product of uranium. The daughter products of radon are solids. Radon occurs coincidentally with any naturally occurring uranium and is therefore one of the common components of the earth's crust. Normal atmospheres contain between 0.05 and 0.2 $\mu\mu c/l$. Because radon is a gas and the daughter products are solids, it is estimated that 95% of the total dose contributed to the lung by radon in equilibrium with its daughters is derived from the daughter product activity.[1, 12]

Chemical symbol:	Rn
Formula weight:	222
Relative vapor density:	7.6 (air = 1)
Radiation emitted:	alpha; average energy 5.5 mev
Specific activity:	
1 μg:	36×10^{10} d/min
At 25° C and 760 mm Hg,	
1 ppm:	0.00908 mg/liter
1 mg/liter:	110.1 ppm

III. Industrial Hygiene Practice

A. RECOGNITION: Radon, a colorless, odorless, noble gas, is found in varying concentrations in most underground workings. Its concentration in mines other than those involved with uranium and radium is generally between 10 and 200 $\mu\mu c/l$. In uranium bearing mining operations, however, the levels encountered generally range from 50–20,000 $\mu\mu c/l$.[6]

B. EVALUATION OF EXPOSURE: An alpha pulse counting ionization chamber (range of 0.1 to 1000 $\mu\mu c/l$), or a direct reading rate meter (10 to 10,000 $\mu\mu c/l$,[5, 6] or 25 to 300,000 $\mu\mu c/l$[7]) may be used. Alpha counting of the daughter particulates may be done.[16] The determination of the polonium content of the urine provides some measure of cumulative radon dosage.[14]

The Disintegration Series of Uranium, Radium, and Radon[16]

Common Designation	Isotope	Principal Radiations	Average Energy[g]	Half-life[f]
Uranium-238	$_{92}U^{238}$	alpha	4.2	4.5×10^9 y
UX_1	$_{90}Th^{234}$	beta	0.2–80% 0.1–20%	24 d
UX_2	$_{91}Pa^{234}$	beta	2.3–80% 1.5–13% 0.6–7%	1.2 min
Uranium-234	$_{92}U^{234}$	alpha	4.8	2.5×10^5 y
Ionium	$_{90}Th^{230}$	alpha	4.7	8×10^4 y
Radium	$_{88}Ra^{226}$	alpha	4.8	1.6×10^3 y
Radon	$_{86}Rn^{222}$	alpha	5.5	3.8 d
Radium A	$_{84}Po^{218}$	alpha	6.0	3.0 min
Radium B	$_{82}Pb^{214}$	beta	0.6	
		gamma	0.3 (0.8)[e]	27 min
Radium C	$_{83}Bi^{214}$	beta	3.1–23% 1.7–77%	
		gamma	1.0 (1.5)[e]	20 min
Radium C′	$_{84}Po^{214}$	alpha	7.7	2.7×10^{-4} min
Radium D	$_{82}Pb^{210}$	beta	0.02	
		gamma	0.05	22 y
Radium E	$_{83}Bi^{210}$	beta	1.2	5.0 d
Radium F	$_{84}Po^{210}$	alpha	5.3	138 d
Radium G	$_{82}Pb^{206}$			stable

Note: e. Number of gamma photons per beta emission
f. Half life in years (y), days (d), or minutes (min)
g. Energy in million electron volts (mev)

C. RECOMMENDED CONTROL PROCEDURES:

(1) *Underground Workings:* Forced ventilation, with the air supplied to the working face, and with abandoned tunnels and drifts sealed off, is necessary.[15]

(2) *Above Ground Operations:* Natural ventilation is adequate, except where quantities of radium bearing materials are stored in enclosed places. Mechanical ventilation is then necessary. Daughter products must be removed with adequate filtration if air is recirculated.

IV. Specific Procedures

A. FIRST AID: None.

B. SPECIFIC MEDICAL PROCEDURES: None.

V. References

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3. DIGIOVANNI, H. J., et al.: USAEC NYC-4616 (1951).
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16. *U. S. Public Health Service Publication ⁂494.* Supt. of Documents, Government Printing Office, Washington, D. C. (1957).

Selenium and Compounds

(Excluding Hydrogen Selenide)

I. Hygienic Standards

A. RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours): 0.1 mg per cubic meter of air.[1] This appears to have been based largely upon the observed effects from hydrogen selenide.

1. *Basis for recommendation:* Clinical experience[2, 4, 9] and animal experiments.[2]

B. SEVERITY OF HAZARDS:

1. *Health:* Moderate and high for acute and chronic exposures, respectively, of the element. Some compounds, particularly the dioxide, the selenites and selenates, are highly hazardous for both acute and chronic inhalation and ingestion exposures. Acute signs in man include sternal pain, cough, nausea, pallor, coated tongue, gastro-intestinal disorders, nervousness, and conjunctivitis.[2, 4, 7, 9, 14] A garlic odor of the breath and sweat may occur. Animal work indicates that prolonged exposure would lead to weight loss and anemia; liver damage has been demonstrated in animals after acute and chronic exposures.[5, 12]
2. *Fire:* Moderate for free selenium.

C. SHORT EXPOSURE TOLERANCE: By analogy to animal data reported[5] for H_2Se, 1 mg/m³ of Se is considered dangerous to life for four hours or more exposure. Deaths in rats have been produced with eight hours exposure to a concentration of 33 mg/m³ of the element.[6]

D. ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE: Unknown.

II. Significant Properties

Pure selenium is a grey, crystalline, semimetallic appearing element of the sulfur group. The compounds of selenium resemble those of sulfur very closely. Like sulfur, it exists in several allotropic forms.

Chemical symbol:	Se
Atomic weight:	78.96
Specific gravity:	4.27–4.80
Melting point:	217°C
Boiling point:	688°C

III. Industrial Hygiene Practice

A. RECOGNITION: Selenium is chiefly used in glass and ceramics manufacture and in production of selenium rectifiers and photocells. Rectifier burn-outs offer large potential exposures, but apparently they are not greatly hazardous.[3]

B. EVALUATION OF EXPOSURE:

1. *Direct instrumentation:* None.
2. *Chemical:* Collection of the dust by impingement or filtration, followed by gravimetric or colorimetric determination.[11]

C. RECOMMENDED CONTROL PROCEDURES: Handle dry powders or molten metal in hood. Exercise care to avoid ingestion of traces of selenium or its compounds while eating or smoking. Skin contact should be avoided.

IV. Specific Procedures

A. FIRST AID: Remove from exposure. Wash skin or eyes immediately to remove splashed material.

B. SPECIAL MEDICAL PROCEDURES: Medical supervision of the workers should be based on general health.[7] Urinary evaluations are also helpful. These may be performed spectrophotmetrically.[8] Normal urine values of unexposed males[15] have been given as ranging from 0–15 micrograms per 100 ml, with an average of 4–5 micrograms per 100 ml.

V. References

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Titanium*

I. Hygienic Standards

A. Recommended maximum atmospheric concentration (8 hours): No value established.

B. Severity of hazards:

(1) *Health:* Low.[1, 4, 7] Titanium compounds introduced into the respiratory tract in physiological solution caused fibrosis of the lungs and bronchi.[6] Implants of titanium metal in muscle tissue proved inert.[3] No occupational case of systemic poisoning has been reported in the literature.

(2) *Fire.* Extreme fire and explosion hazard when in powdered or dust form.[5] High fire hazard in the form of fine chips or turnings. Dust clouds may self-ignite and explode if the mean particle size is less than ten microns. Low energy ignition source required for ignition of dust clouds of larger particle size. Lower explosive limit may be as low as 0.045 ounces per cubic foot and pressures of over 80 psi may be attained. Ingots, bars, rods, etc. are relatively nonflammable. Contact of titanium and its alloys with strong oxidizing liquids is potentially explosive.[2] Titanium powders should be stored in the presence of at least 20% water by weight.

C. Short exposure tolerance: Unknown.

D. Atmospheric concentration immediately hazardous to life: Unknown.

II. Significant Properties

Titanium is a gray metallic solid and may be received in massive forms such as ingots, bars, rods, etc., or as a powder.

Chemical symbol:	Ti
Atomic weight:	47.90
Specific gravity:	4.5
Melting point:	1800°C
Solubility:	Soluble in dilute acids

III. Industrial Hygiene Practice

A. Recognition: No identifying characteristics of taste, odor, or irritation. Its oxides are used extensively as a pigment in paints and for welding rod coatings. Titanium metal is used in alloys and in its massive forms by the Atomic Energy Commission.

B. Evaluation of exposures:

(1) *Instrumentation:* None available.

(2) *Chemical:* Determination by spectrographic procedures is recommended.

C. Recommended control procedures: Control dust production with water wash dust collectors. Ducts should be short, straight sections with conveniently spaced clean-out doors. Dust collectors should be separate from other ventilation systems. Machining should be performed in the dry state or with the use of high flash neutral mineral oil coolants. Soluble or vegetable oil coolants should not be used. Safety goggles or face shields, smooth fire resistant clothing without pockets, cuffs or other recesses, and leather gloves should be worn. Trousers should be worn over high top leather shoes. Safety showers should be provided. Only approved graphite type (such as G-1 powder) or salt-resin base (such as Met-L-X) extinguishing agents for metallic fires should be used.

IV. Specific Procedures

A. First aid: Eyes should be irrigated by large amounts of water for 15 minutes followed by medical attention. Titanium which has penetrated the skin should be removed before dressing the wound.

B. Special medical procedures: No special requirements for placement or treatment appear necessary.

V. References

1. Air Foundation of America, Inc. (Industrial Hygiene Foundation), Abstract No. 217 (1937).
2. Ambrose, P. M., et al.: *Bur. of Mines Inf. Circ. 7711* (1955).
3. Beder, O. E., and G. Eade: *Surgery 39:* 470 (1956).
4. Fairhall, L. T.: *Industrial Toxicology.* 2nd Edition. The Williams & Wilkins Co., Baltimore (1957).
5. Hartmann, I., J. Nagy, and M. Jacobson: *Bur. of Mines, Rept. Invest. 4837:* (1951).
6. Magileuskoya, O. Ya, et al.: *Ind. Hygiene Digest, No. 1412* (1957).
7. Queries and Minor notes: *J. Am. Med. Assn. 146:* 1175 (1951).

* Except where specifically indicated, this Guide applies to elemental titanium.

Because of space limitations, it is impossible to list all methods of exposure evaluation. The selections have been made on the basis of current usage, reliability, and applicability to the usual industrial type of exposure. Any specific evaluation and/or control problem will involve professional judgment. This can best be done by professional industrial hygiene personnel.

Respiratory protective devices are commercially available. Their use, however, should be confined to emergency or intermittent exposures and not relied upon as primary means of hazard control.

A relative scale is used for rating the severity of hazards: nil, low, moderate, high, and extra hazardous.

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News of Local Sections

Western New York Section

At the April meeting J. F. Parsons, illuminating engineer for the Niagara Mohawk Power Corporation, and Elizabeth Pierce Olmstead, M.D., ophthalmologist, discussed industrial lighting from the engineering and medical aspects. This made a most interesting presentation. At the business meeting, the following officers were elected: Herbert I. Miller, Jr., Bethlehem Steel Company, President; Richard F. Scherberger, Eastman Kodak Company, President-Elect; John T. Murray, Western Electric Company, Secretary-Treasurer; and Lewis Fancourt, Mine Safety Appliances Company, Director.

Michigan Sections

The Michigan (Detroit), Northeastern Michigan and Western Michigan Sections joined forces for their May meetings. A full day's session was held on May 19 at the Henry Ford Museum, Greenfield Village. The program and speakers were: A Technique for Paint Spray Booth Effluent Evaluation—J. C. Radcliffe, Ford Motor Company; Methods of Calibrating Field Instruments—H. E. Jaworski, Detroit Bureau of Industrial Hygiene; Unusual Industrial Hygiene Problems—B. D. Bloomfield, Michigan Division of Occupational Health; Radiation Exposure and Workmen's Compensation—J. H. Sterner, M.D., Eastman Kodak Company; Dow Experience with Davis Halide Meter—A. W. Schaffer, Dow Chemical Company; A Year's Experience with Radiation Regulations—J. C. Soet, Michigan Division of Occupational Health. Those in attendance also toured the Henry Ford Museum.

New Jersey Section

Sixty-two attended the February meeting, including several local ventilation engineer guests. John Kane of American Air Filter was our speaker and his able presentation was entitled "Balanced Ventilation—Its Problems and Possible Solutions." Several interesting case histories pertinent to the subject were discussed.

Election of officers, 1959–60, was held at this meeting. Officers are: President, F. W. Church, Esso Research and Engineering Company; President-Elect, K. R. Doremus, Merck and Company; Secretary-Treasurer, Jane W. Vosecek, New Jersey Department of Health; Director (3 years), G. D. Ware, Western Electric Company; Director (1 year), A. R. Jones, Esso Research and Engineering Company.

Ohio Valley Section

The March 24th meeting was held at the Cincinnati Milling Machine Company, Norwood, Ohio. A fine dinner, compliments of the Company, was provided and a tour of the plant followed. Dr. E. C. Binstadt spoke on the industrial hygiene problems encountered in the operations of the Cincinnati Milling Machine Company.

Applications from seven new members were approved and the results of the election of officers were announced. New officers for 1959–60 are: Ralph F. Hoffer, General Electric Company, President; Dohrman H. Byers, U.S. Public Health Service, President-Elect; Robert H. Starkey, National Lead Company of Ohio, Secretary-Treasurer; and Robert G. Keenan, U.S. Public Health Service, Director.

Georgia Section

The General Motors Corporation, Buick-Oldsmobile-Pontiac Division, Doraville, Georgia was host to the Georgia Section at their May meeting. A dinner meeting was followed by a tour of the plant.

The Section is making a drive for membership and has distributed information on proposals of the AIHA-ACGIH Joint Committee on Certification.

Personnel Notes

Dr. Charles E. Lewis has been appointed plant physician of the Texas Eastman Company, Longview, Texas.